

VOLUME VIII

JANUARY, 1923

NUMBER 1

JOURNAL

OF THE

American Association of Cereal Chemists

OFFICIAL PUBLICATION OF THE
AMERICAN ASSOCIATION OF CEREAL CHEMISTS

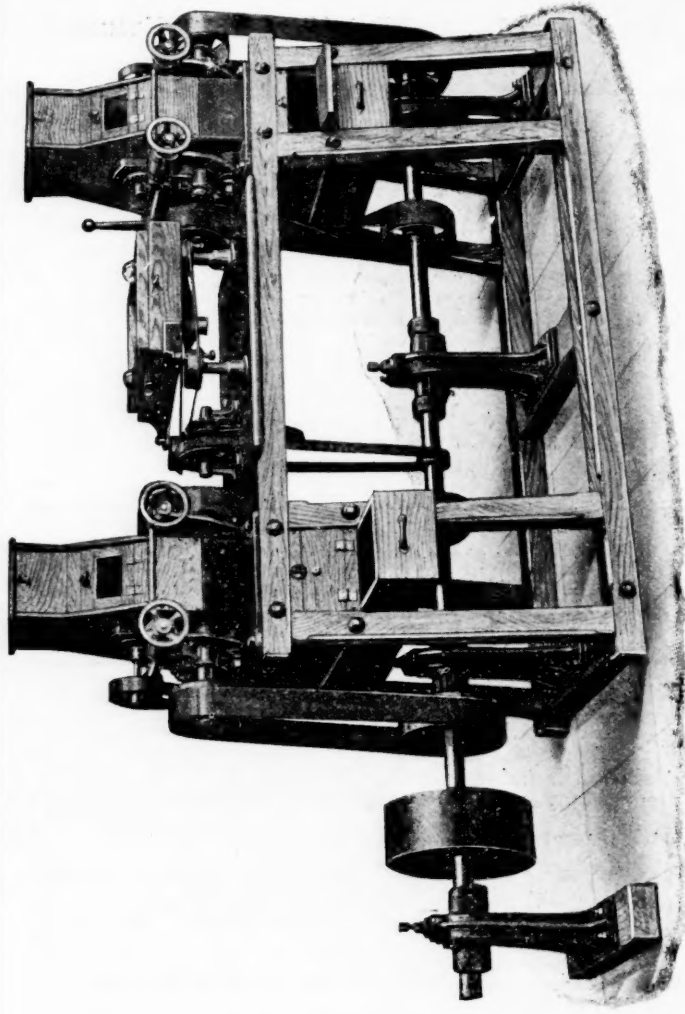
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PUBLISHED QUARTERLY
AMERICAN ASSOCIATION OF CEREAL CHEMISTS
HUTCHINSON, KANSAS

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Shortening Period of Digestion in Protein Determinations On Wheat and Flour, By the Aid of Perchloric Acid.

By ROY K. DURHAM.
Chemist, Rodney Milling Co., Kansas City, Missouri.

With the advent of the practice of selling and buying wheat upon a basis of protein content, comes a demand for shorter methods of determining that constituent. Control chemists are also seeking means to shorten time required for analysis of raw and finished products. This demand furnished the incentive for the work here presented.

Mears and Hussey's recently published article on "The Use of Perchloric Acid as an Aid to Digestion in the Kjeldahl Nitrogen Determination," suggested the possibility of the use of that reagent in the cereal laboratory. Accordingly the following experiments were made:

For preliminary tests, a sample of flour of unknown protein content was employed. A series of determinations were made to determine the minimum time required for complete digestion, using official Kjeldahl-Gunning method and heating over 110 volt Gilmer electric heatres. Previous tests had led to the conclusion that 110 volt heaters supplied a higher temperature than those of 55 volts connected in series. All determinations became clear in 22 or 23 minutes, when starting with heaters perfectly cold, or 15 minutes when using heaters while yet hot from previous digestions. Tests were made allowing digestion to continue for periods of 10, 20, 30, 40 and 50 minutes after clearing. Four checks were made on each period. Table No. 1 shows results.

TABLE NO. 1.

Period minutes	Percent protein
10 minutes	11.73
20 minutes	12.04
30 minutes	12.14
40 minutes	12.20
50 minutes	12.18

That period of 40 minutes after clearing, or 63 minutes from time heaters were turned on, was selected as minimum time that should be allowed for complete digestion.

Experiments were then made on the same flour, by the same method as before, but modified by the addition 1, 2, 3, and 4 c. c. of 60% Perchloric acid. Where 1 and 2 c. c. respectively were used, 15 or more minutes were required for determination to become clear, and these were eliminated.

Those receiving 3 and 4 c. c. cleared in 5 to 7 minutes. The addition of 3 c. c. caused them to clear just as fast as with 4 c. c. It was thought that, though 4 c. c. did not shorten the time of clearing any more than did 3 c. c., there might possibly be a material saving on the time of complete digestion. Accordingly, as before, tests were made on these two groups to determine

minimum time required for digestion. Sub-groups of 4 each were digested 10, 15, 20 and 25 minutes.

TABLE No. 2.

3 c. c. HClO_4 .			4 c. c. HCl .		
No.	Time of Digestion	Percent Protein	No.	Time of Digestion	Percent Protein
1		None	1		8.92
2	10 min.	6.44	2	10 min.	None
3		6.24	3		None
4		None	4		None
5		11.48	5		9.32
6	15 min.	11.64	6	15 min.	9.56
7		9.24	7		None
8		11.40	8		None
9		12.20	9		None
10	20 min.	4.28	10	20 min.	11.96
11		12.16	11		None
12		12.16	12		12.04
13		12.20	13		None
14	25 min.	12.12	14	25 min.	12.04
15		12.12	15		12.12
16		None	16		None

Results proved conclusively that best results were obtained using 3 c. c. of Perchloric acid and digesting 20 minutes. A loss of all or nearly all of the Nitrogen in 9 out of 16 determinations treated with 4 c. c., eliminated all hope of increased benefit by using larger amounts of Perchloric acid.

Further observations were made using 3 c. c. and digesting 20 minutes in an effort to ascertain cause of frequent loss of a considerable amount of Nitrogen. All changes were folded in a 10 c. m. filter paper and dropped into flask in order to prevent any material from adhering to the neck. Sodium sulphate, copper sulphate, sulphuric acid and perchloric acid were added in the order named. The flasks were only gently shaken. In some cases the filter paper decomposed at once, in others it remained on top the acid until heat was applied. It was observed that in the case of the latter described determinations, a clear solution resulted in 3 or 4 minutes, but a part of the change remained in a small lump darting about on the acid's surface like a piece of metallic sodium on water. This lump would suddenly disintegrate and solution would become cloudy and finally clear up again in about 7 minutes. In every case, it was these determinations displaying this phenomenon that showed an appreciable loss of nitrogen. Following these

observations, all changes were thoroughly mixed with the acid before starting digestion and no further trouble was experienced.

Subsequent tests on more than 100 wheat samples taken at random have analyzed, when treated with Perchloric acid, only .02% less protein than when official Kjeldahl-Gunning method was used.

SUMMARY.

Some chemists report a 20 or 25 minute digestion sufficient for accurate results. It is possible they, too, may be able to shorten that time somewhat by the use of Perchloric acid. Though it may seem too small a saving to be worth while, it should be remembered that it will be the aggregate of such small savings that will eventually reduce to a few minutes, the time required for a protein test. It is certain that in laboratories where 50 minutes to an hour is found to be the minimum time of digestion, a very material saving of time will result by the use of 3 c. c. of 60% Perchloric acid in addition to the reagents called for in the official methods, provided charge and reagents are properly mixed before starting digestion.

Value of Ash Determination.

By S. J. LAWELLIN.

Chief Chemist, Milton Hersey Co., Winnipeg, Manitoba, Canada.

Many and varied are the interpretations placed upon the determination of ash in flour. There can be but three views to the subject. Those of the miller, the baker and the chemist, as they are the only ones interested. People are always skeptical of and undervalue anything that they cannot readily understand. We will not attempt to outline the views of the miller and baker but will give you the value a chemist places on the determination of ash, why he wants it and to what use he puts this determination. We will also give our ideas of the way in which the miller and baker should use the reported value for ash in any particular flour.

No chemist who has been well trained, or who knows anything about flour, will place any value upon a certain flour from the percentage of ash alone. The first thought that the ash figures bring to him is an indication of the grade of the flour. It is quite true that the grade or percentage will be greatly indicated in a flour by the figures for ash but results for other analyses must bear out this fact before one can even say approximately what grade the flour is. However, ash is of importance, when part of a complete analysis, in judging the grade, value, and degree of milling of any flour. Ash is one of the features of flour which can be kept absolutely uniform in the mill where a laboratory is maintained. The other features possible to keep uniform are protein and moisture.

The daily routine of ash determinations indicates to the chemist how the mill is running. He is, in a way, the watchman for the miller. Should

any flour stream go astray, any machine fail to function properly, or any irregularity occur in the mill it will be reflected at once in the ash content of the flour. When any variation occurs it is the duty of the chemist to immediately notify the miller, giving any other information the percentage or appearance of the ash may show. There is always a slight variation, even in duplicates, in any ash determination. The limit of variation should never be more than 0.02% and this may be either plus or minus. When duplicates are run on a sample and the results do not agree as close as 0.02% there has been some mistake of the chemist. When duplicates come within this limit of error and the difference from the daily average is more or less than 0.02% the mill or miller is to blame. In case of the chemist's mistake a recheck should be run and in the latter case the mill should be gone over carefully.

It is our opinion that there are mighty few trained chemists who would make the absurd statement that ash is a real indication of the quality or baking value of a flour. But there are many, so called, "short-course" men and men who have worked in a laboratory as helpers who might make such statements. However, they generally compare to a chemist about the same as a sweeper would to a master miller. It would take a rather complete analysis of a flour to cause a chemist to pass judgment as to the quality or value of a flour. This analysis should contain ash, moisture, total protein, soluble protein, soluble solids, and color and should be supplemented by figures for gliadin and glutenin percentages. From these the trained chemist could form a fairly accurate estimate of the grade, degree of milling, quality and baking value of a flour. But even this analysis should be supplemented with a baking test, either experimental or commercial.

However, to say that anything definite can be told by the ash test alone is ridiculous. It would only give one an indication. One could use the ash test to good advantage as one of three variables. If the ash and flour percentage are known it will give one a very good idea as to yield and as to whether the milling has been good or bad. If one knows that the flour has been milled properly and has the percentage of ash, then it will give an excellent indication of the percentage of the flour. But judge the absolute value of a flour by the ash alone is out of the question. And right here let us say it is unfair to compare the ash content of any two flours unless taking into consideration their moisture content. The only fair comparison would be at the same moisture content or on a moisture-free basis. The trained man will run a moisture test on each and every flour on which he runs an ash test and will weigh them at as near the same time as possible. In reporting the ash on any sample he should also report the moisture so that anyone can make comparisons. It is always best, when comparing two samples of flour for ash, to first calculate them to a moisture-free basis. This can easily be done by means of the formula $P_2 = P_1$ divided by $1 - M$ where P_2 equals percentage ash on the moisture free basis, P_1 equals per-

centage ash at known moisture content and M equals known moisture content. Of course the calculation to a moisture free basis will make the percentage of ash somewhat higher. Take for instance a flour with an ash of 0.40% and a moisture content of 12.00 %. Calculating this to a moisture-free basis would make the ash percentage on the dry basis equal 0.455%. This should be taken into consideration as it does not represent the percentage of ash of the natural flour.

For their guidance the American Association of Cereal Chemists has set a standard for moisture content of flours. This standard is 13.50% and is to be used in comparison of natural flour coming from the mill. This figure was chosen on account of its being the nearest average to the moisture content of the flour when packed and as it is the maximum moisture content allowable under the Pure Food and Drug Act. Taking the sample previously noted as having 0.40% ash and converting this to 13.50% would give us the value 0.393%. This value is obtained by the formula $P_2 = P_1 \times (1 - M)$, where P_2 equals percent ash at 13.50%, or any desired moisture content, P_1 equals percent ash on dry or moisture-free basis and M equals 13.50% or any desired moisture content.

In order to make intelligent use of ash percentage one must have some idea of the kind and class of wheat used. Spring, Winter, hard, soft, dark, semi-dark and yellowberry wheats would not necessarily give identical ash figures on the same percentage patents at the same yield. Flour is bought and sold very much according to the flour classes, winter or spring and hard or soft wheat flour. Even in the mill using many kinds and classes of wheats the increase or decrease of any particular wheat may make a material difference in the ash content of the daily run of flour. Of this the chemist and miller must take account. It is a mistake for the chemist to not know at all times the exact percentage composition of the mill mix, the yield of flour and feed, the tempering conditions and how the mill is grinding on the mix and under these conditions. It is also a mistake for the miller to not know everything going on in the laboratory and all the characteristics noted there. The chemist should know these things of the mill in order to be the better able to interpret his ash results and by knowing the laboratory the miller gets more value from the chemist's results.

When burning ash the chemist knows on which leaf his rheostat should be set to get the best muffle temperature and the best burn on his ash. He does not go by figures alone but notes the color of his ash, the manner in which it has burned, and the flakiness and smoothness. These things he takes into consideration when passing judgment from the ash determination. He even has learned many things from so small a matter as the length of time required to get a good clear ash at the proper temperature. He knows that to get good standard comparable results on routine ash work that it must be done under standard conditions and at a standard temperature. Ash that

is black or dark colored, glazed, or fused is improperly handled. Properly burned ash will be flaky and the color will range from a white gray to medium dark gray. The ash will not lie flat in the bottom of the crucible but will be burned away from the sides and have much the form of an inverted cone. It should be uniform in color throughout and feel smooth to the fingers. When exceptions are noted the chemist knows that there is a reason and looks for it.

The miller's use of the ash results should be in checking the operation of his mill from day to day. He should know what effect the fineness of his breaks and reductions would have on his ash and be able to make deductions accordingly. Even the corrugations on his rolls have a certain influence. In the operation of his bolters, sifters, reels and purifiers the miller can greatly influence his ash. The kind and size of cloths he uses are of material importance. The point of suction, as also the amount, in his dust collecting system should be studied by the miller for its influence on his product. The ash and color together will show him quicker and more satisfactorily than anything else the non-functioning of these vital points in his system. He should be able to forecast the effect on his ash and color of any change in his wheat mix or milling system. The miller should also be able to take the chemist's results for ash and know where to look for trouble in the mill if any has been indicated.

We do not believe that many millers use the percentage of ash as an argument for the quality of the flour. Our experience has been that they use it more as an argument that they are turning out uniform flour and the specific patent called for on any contract. Some may argue that their low ash content is a mark of the quality of their flour but they are the exception and it is a serious mistake. However, many millers do use, and are justified in using, the percentage of their ash as an indication that they are doing quality milling, honest milling, and giving the customer the kind of flour that he purchased. The miller who mills to a color standard and to a certain protein and ash standard will certainly produce a uniform quality flour. For the miller to talk flour quality from ash percent alone is quite absurd. He may use it, but again not alone, in talking uniformity, grades, or even milling quality but the mill with an unassailable reputation need not talk milling quality. Then as far as a sales argument is concerned there is left to the miller only two uses for an ash determination, grade or percentage of flour and uniformity. When talking uniformity based only on the ash determination he should then only mention uniformity of ash content as there are too many other features entering into what would be called an absolute uniform flour. The miller's great use for ash determinations are for his own information in the control of the mill and here it becomes of great importance. If the miller will do a little experimenting there is no

limit to the use to which he can put the ash determination. And when it comes to experimenting the chemist is with him, heart, soul and body.

The baker is the one who gets the least value from, and makes the most fuss over, an ash determination. It would seem that they have some idea that the percentage of ash has some direct relation to the quality of the flour. Just what this relationship is would be hard to say. It is certain that there is no definite connection between the percent of ash and the quality of flour. It is quite true that with wheat of the same kind and class a flour of a certain quality and grade will somewhat tend to vary in quality as the ash varies. To what extent this can be carried has never been determined but is believed to lie within very narrow limits. Perhaps when one says "patent flour" to a baker he has some definite idea or standard for the ash of that flour but we must consider, however, that patents may be anywhere between 65% and 80% with no very definite lines at those percentages. Also, we have the baker's patents at anywhere from 70% to 95% so the word patent is quite meaningless and could not be coupled with any definite percentage of ash. To buy flour by ash content alone would certainly be overlooking the important points. Naturally in buying his flour the baker should know the ash content and should use it as an indication of grade only. The percent of ash would show no appreciable effect on the quality of the flour or the bread baked therefrom.

Knowing the ash content of the flour he specified the baker has every reason to expect flour with an ash content that will not vary over 0.02% from that figure. If he is afraid the miller will ship him a cheaper flour he certainly has the privilege of having his flour analyzed to see if he is getting the grade he purchased. But does he take into account the lowered moisture content occasioned by transportation and storage? If not he is doing the miller an injustice. To condemn a flour on the percent of ash alone is an injustice to any flour. The baker should not buy flour on ash content alone nor should he specify a certain percentage of ash. The baker would do well to purchase flour of good color and dress and specify protein content, that will give him a standard loaf of bread in a certain fermentation period. This flour to be sound and of grade specified. But that is several years ahead of the average baker.

Summing it all up we would say that the chemist's first use of the ash test is an indication of the grade. Coupled with the characteristics noted during burning he uses it to judge kind and class of flour and taken in connection with a complete chemical analysis to judge the quality of milling and the grade, percentage and quality of the flour. However, to pass final judgment from the ash test alone is going too far.

The miller's main use of ash is in the operation and control of his mill, to assist in making his mill mix, and as an indication of the quality of

his milling. He is undoubtedly the one who gets the most value out of the ash determination.

The baker makes use of the percentage of ash as an indication of the grade of the flour. He may also use it to check flour shipments and see that he is getting the grade he purchased but this is only when he is suspicious of the miller and does not often occur. To purchase flour on ash content alone is certainly a poor business system.

The ash test is of importance but not in determining flour quality or value. Its greatest importance is when supplemented by a complete chemical analysis and to fail to make allowance for variation of conditions of determining ash and variation of moisture content is to fail to be fair.

Bleaching and Maturing of Flour.

By DR. F. L. DUNLAP.
Consulting Chemist of the Industrial Appliance Company.

When your president, Mr. Lawellin wrote to me some time ago, asking if I would discuss this subject before the members of your Association, I answered him in the affirmative. I had some doubt in my mind as to just what differentiation he made between "bleaching" and "maturing." This doubt has not been resolved, for I have not asked him. He possibly wished me to do this for him and to tell you how I personally view the matter. This I am glad to do. Whether I strike a responsive chord or not, remains to be seen.

I think we all are indebted to Dr. Morrison of the American Institute of Baking, who is now publishing in Baking Technology, the new journal of the American Bakers' Association, a resume of flour treating methods, under the caption of "Why Bleached Flour?" For the first time, we have quite a complete resume of the literature readily accessible. I have been forcibly impressed as I have read Dr. Morrison's publication, from month to month, together with my own general knowledge of the subject, how few of the methods that have been proposed really have been of any particular moment in a practical and operative way, and have achieved anything like a commercial success.

So far as a practical adoption by millers is concerned, the forerunner of all the methods is what we know as the nitrogen dioxide process. You are all familiar in a general way with this process as now conducted. Its operation depends on the heat generated by an electric discharge causing some of the oxygen and nitrogen of the air to combine, with the formation of nitric oxide. The chemical substance then takes up oxygen from the air

and forms nitrogen dioxide. It is this latter that is the active agent in this process for treating flour.

This method of treating flour with NO_2 is the earliest of the commercial methods used in the United States, and the flour so treated was known as "bleached flour." The method and product produced by such method has been the source of extensive litigation both in this country and in England. Nitrogen dioxide treated flour was the one which became more or less generally known through government activities, for at that early period, it was the sole treated flour on the American market. Published results of experiments both in this country and abroad, indicate that flour treated with nitrogen dioxide, while very much whiter than before treatment, is not materially affected in its baking qualities.

While the term "bleach" from a dictionary standpoint does mean "to make white," yet with this historical setting, it has, at least to me, come to mean, the treatment of a flour whereby such flour is made whiter, but nothing else.

There is little need, in such an audience as this, to go to any lengths to discuss the aging of flour. Flour when it ages, turns whiter and increases in the quantity of acid. As flour ages, it becomes a better flour, for it produces a larger and better loaf of bread. Freshly milled flours do not produce the best of which they are capable. A great advance was made in the milling art, by the introduction of a method for treating flour in the mill, whereby such freshly milled flour at once took on the properties of a properly aged flour, so that the flour could then go to the consumer in condition to render at once its highest baking value.

The aging of flour is not commercially feasible. The cost is against it, for one thing. Another objection, from a commercial standpoint, is the impossibility of following the condition of the aging flour, especially if one wishes to catch it at its peak, for stored flour is constantly varying in its baking capacity, finally reaching an optimum and then beginning to decline. So that any method which the miller can employ, assuming for the moment that it is unobjectionable from a health standpoint, which will instantly convert a flour to its optimum baking value and then stabilize it, is of great moment to the public.

This state of affairs is brought about in flour by treating it with chlorine and the general result is known as "maturing," i. e., the general effect of "maturing" flour is that which nature produces in aging flour—a whitening effect together with greatly improved baking qualities. You now can get what is the distinction between "bleached" and "matured" flour.

Whatever the method used to treat flour, if the resultant product is whiter, then we know that the yellow coloring matter of the flour has been discharged to a greater or less extent. There is not a great amount known

concerning this yellow coloring matter of flour, but it will be interesting, I think, to discuss with you just what we do know.

The yellow coloring matter of flour is *carotin*. It was not until 1909 that we find a reference to the character of this color in flour. In that year, Wesener and Teller † stated that "we have isolated the coloring matter and have identified it, or at least have established a very close relationship with other natural vegetable colors." In 1911, in another article ‡ they say, "in a previous article we called attention to the fact that the coloring matter of flour is of the same nature if not identical with carotin * * *." While they state that they have evidence of the identity of this coloring matter of flour and carotin, which is to be published subsequently, yet I have been unable to find anywhere in the literature that such evidence has ever been brought forward. Wesener and Teller, are, however, the first to mention that carotin is the yellow coloring matter in flour.

Carotin was first isolated from carrots in 1831 by Wackenroder. Zeise in 1847, described it more completely and gave it the formula C_5H_8 . In 1861 and 1867, Husemann carried out more intensive research and gave it the formula $C_{18}H_{24}O$. The work of Arnaud led him to the conclusion that carotin was a hydrocarbon. In 1885-1887, Arnaud investigated a yellow crystalline substance accompanying chlorophyl and he discovered that this was probably identical with carotin, the coloring matter of carrots. Hensen's experiments in 1889 were in accord with Arnaud's and this was further verified in 1893 by Montverde. The only analysis of carotin from leaves had been given by Immendorf and there was no assurance of the identity of carotin from leaves and carrots.

Arnaud's analytical data for carotin, and an iodide prepared from it, led him to give the formula as $C_{26}H_{38}$. Up to the time of Willstatter and Miegl in 1907, Arnaud's results were never questioned. He worked out a very beautiful colormetric method for determining carotin in the green portions of plants. He found that petroleum ether dissolves only one yellow coloring matter from plants, namely carotin.

The final settlement of the identity of carotin in leaves and carrots is due to Willstatter and Miegl §. Both by analyses and by molecular weight determinations by the boiling point method in both $CHCl_3$ and CS_2 , they determined that this substance was a hydrocarbon $C_{40}H_{56}$. In keeping with this, they prepared a beautifully crystalline iodine addition produce $C_{40}H_{56}I_2$ containing 32.5% iodine.

Willstatter and Miegl say that the extremely wide distribution of carotin would suggest that it has some extremely valuable physiological function

† Jour. Ind. Eng. Chem. 1,702 (1909).

‡ Ibid. 3,917 (1911).

§ Ann. 355, 1 (1907).

in plant life. Concerning this function, there are two hypotheses. (1) That it has a function in the assimilation of CO_2 . (2) That its function is in some way connected with the oxygen liberation of plants together with the CO_2 absorption or assimilation. Willstatter and Mieg thinks that carotin acts rather as an oxygen carrier.

Carotin forms red crystals of a coppery luster, having a melting point of 167.5 degrees—168 degrees C. In powdered form it looks like cinnabar. It dissolves in concentrated H_2SO_4 with an indigo blue color, which on dilution, precipitates in green flocks.

Willstatter and Mieg say that carotin absorbs oxygen and is thereby completely decolorized. The oxidation goes on rapidly even in the dark and in the absence of moisture.

If powdered carotin is placed in a desiccator and oxygen introduced, in about three weeks constant weight is reached and the carotin had taken up 34.3% of its weight of oxygen, i. e., this corresponds to the taking up of over eleven atoms of oxygen.

So far as I am aware, the only scientific evidence which has ever appeared in the literature, showing the identity of the yellow coloring matter of flour and the carotin from other sources, appeared in 1912, in a "Report to the Local Government Board by Dr. G. W. Monier-Williams, on the nature of the coloring matter of flour and its relation to processes of natural and artificial bleaching." In his report Monier-Williams refers to this product in flour as "carotene." He says, "A photograph of the absorption spectrum of pure carotene in petroleum, shows three well defined bands in the blue and violet regions at approximate wave lengths of 4760, 4470 and 4200, and a suggestion of a fourth band at 3950 * * *. A comparison of the absorption spectrum of a petroleum ether extract of flour with that of pure carotene in the same solvent, indicates that the coloring matter is either identical or closely related, the position of the bands in both cases being practically the same."

In his experimental work, Monier-Williams did not work with carotin isolated from flour, but with carotin obtained from carrots. He does, however, give three figures on the amount of carotin in patent flour, figures determined by photographic and colormetric methods. One sample contained 1.3 and 1.4 parts of carotin per million, and the other 2.0 parts. Of course, these figures would vary considerably, depending on the grade of the flour and the character of the wheat from which the flour is milled, but the amount of carotin in any event, is not very large.

In one set of experiments, Monier-Williams exposed 0.1—0.2 gram samples of carotin to the air for two months, in both the city of London and in Widnes. These increased in weight about 30% and became lighter in color, i. e., a light orange red. The increase in weight was at first very slow, being hardly perceptible for 6 days, but after this, it became fairly rapid.

In one sample, equilibrium was reached in 75 days with 32.3% "oxygen absorbed." This corresponds fairly well with the 34.3% oxygen absorption obtained by Willstatter and Mieg, in treating carotin with pure oxygen.

An interesting observation of Monier-Williams on these exposed samples, is that the smallest trace of nitrite could not be detected in any of these carotin samples, by the Griess-Ilosvay test, even after the carotin had been submitted to long exposure to the atmosphere, nor could any nitrogen be found by the Kjeldahl method on a 0.2 gram sample using salicylic acid and sodium thiosulphate. He remarks that "it is only reasonable to assume that the natural aging of flour is a similar process." I judge that Monier-Williams means that the whitening of flour through aging is due to the oxidation of the carotin.

He showed that carotin combines or reacts readily with NO_2 , and also states that flour exposed to the air takes up nitrite reacting material or such nitrite reacting material is produced within the flour itself. Would not the influence, be a fair one from Monier-Williams, and Willstatter and Mieg's results, that in all likelihood nitrite reacting material plays little or no part in the whitening of flour on aging, but that oxygen plays the major role? Be that as it may, we have Wesener and Teller stating with respect to the use of NO_2 , that "evidently the artificial aging of flour is identical with the natural aging which has been going on ever since white flour was produced."

Notwithstanding the fact that the whitening of flour on aging is now generally accepted as being produced through chemical changes taking place in the carotin, occasionally we find wierd ideas expressed as to its cause, as for example those of an anonymous writer in the *Chemiker-Zeitung* last November. This writer expressed the views that this phenomenon of the whitening of flour on aging was due to the formation of white fatty acids.

Before leaving this general discussion of carotin, I believe you will find of some interest, a brief reference to an article published in the *Journal of Biological Chemistry* in 1916† by Wells and Hedenburg, "On the Toxicity of Carotin." In this work they compared the relative toxicity of carotin, $\text{C}_{40}\text{H}_{56}$, with dischlorcarotin, $\text{C}_{40}\text{H}_{56}\text{Cl}_2$. Their statement in this point is as follows:

"Evidently carotin possesses no considerable toxicity if any, and chlorination does not make it demonstrably toxic. The quantities of carotin used in these experiments are enormous in comparison to the amounts that could be obtained from any food, even carrots. The larger doses used, 0.2 grams, is as much as is contained in 200 kilograms of flour * * *."

"Although the absence of either symptoms or anatomic evidence of irritation after intraperitoneal injection of carotin, indicated that it can have

† Jour. Biol. Chem. 27,213 (1916).

little or no local effect, this was determined more definitely by a series of intradermic tests * * *. These experiments indicate that even in relatively very large doses, carotin, whether in its actual state or saturated with chlorine, is almost entirely devoid of toxicity. Such large amounts as 20 mg. injected intradermically cause only a local edema and inflammation, but no necrosis.

The extensive studies of Palmer and Eckles[‡] indicate that carotin is almost universally distributed throughout all animal bodies, coming chiefly, if not solely, from the food. Our few experiments seem to be sufficient to warrant the assumption that any such quantities as can even accumulate in the tissues have no harmful effects."

So far as the action of gaseous chlorine on flour is concerned, I think we may state the broad principles of such action by saying that it acts on the carotin and unsaturated glycerides, forming addition products. We know further, that water and chlorine react, this reaction being carried on until an equilibrium is established between the HCl and HClO formed and the H₂O and Cl. Hypochlorous acid would, of course, be readily reduced with the formation of hydrochloric acid. The equilibrium would then be re-established with subsequent reduction of the HClO and so on, until the chlorine has been converted into hydrochloric acid. It is the hydrochloric acid which gives the increased acidity to flour which is treated with chlorine, and this increase is capable of control by the degree of treatment.

There are also other interesting changes which take place in flour which has been subjected to this action. Among these is one which acts so as to maintain the acidity of the treated flour at a constant point. In this way we have a marked differentiation from untreated flour, the acidity of which keeps constantly increasing. In as much as the intensity of the acidity is probably the most important variable in determining the baking value of any flour, it will be of interest to you to give an example of results obtained from fresh flour and the same flour subjected to treatment with chlorine. The sample in question was a spring wheat.

FLOUR	TIME	ASH	ACIDITY	VOLUME
Fresh	at	0.40	0.112	2060
Matured	once	0.40	0.151	2340
Fresh	After 116	0.40	0.133	2300
Matured	Days	0.40	0.151	2350
Fresh	After 259	0.40	0.135	2390
Matured	Days	0.40	0.151	2350

From these results we can readily see that the fresh flour had nearly reached its optimum in baking capacity whereas the matured flour remained

[‡] Jour. Biol. Chem. 17,191 (1914).

constant. The greatest change in the baking value of the fresh flour developed after 4 months of storage, but the baking value continued slowly to increase up to the 9th month and had not yet begun to recede.

On the other hand a sample of fresh flour in storage gave these results: The volume of the loaf was, when the flour was baked at once, equal to 2050 cc. After 117 days this volume had increased to 2440 cc., after 308 days the volume had decreased to 2250 cc. The difference in the time it takes a natural flour to age to its optimum value, is undoubtedly not only influenced by storage conditions, but by the different blends of wheat from which the flour is produced, the method of tempering, as well as the manner of milling.

In this connection it is interesting to note a reference to some, as yet unpublished, work of Bailey and Johnson, which is found in the concluding installment of Dr. Morrison's article on "Why Bleached Flour?"§ "Bailey and Johnson (1922) have studied the effect of chlorine bleaching upon the electrolytic resistance and hydrogen ion concentration on water extracts of flour. They found that bleaching flour with chlorine increases the specific conductivity, hydrogen ion concentration, and buffer action of flour extracts in direct ratio to the quantity of chlorine used. These differences apparently do not disappear on storing the flour for some months."

This stabilizing of the baking quality of a matured flour, which baking quality depends so largely on the hydrogen ion concentration, is undoubtedly due to the action of the chlorine on the enzymes normal to the wheat berry, whereby their activity is arrested. This raises the question as to what the increase in acidity of a stored flour is due. So far as we now know, the acidity of flour is due to phosphoric acid or its acid salts, and to amino acids; the former are doubtless of more importance from the standpoint of acidity. The source of the phosphoric acid or its acid salts is phytin, which undergoes hydrolytic cleavage through the activity of the enzyme phytase. Proteolytic enzymes would hydrolyze the proteins, with the formation of amino acids as a final cleavage product. Proteolytic enzymes do not all have the capacity to hydrolyze proteids to the extent that amino acids are formed. For example, pepsin causes a hydrolysis of proteids with formation of preteoses and peptones, which are only an intermediate step toward the amino acids.

The arresting of the activity of the enzymes in flour by the action of chlorine is not a serious matter. The enzymes that are present, are there for the sole purpose of acting when the wheat berry is utilized to produce its kind. Then these enzymes become necessary to split the contents of the berry into suitable food, for nourishment of the growing embryo, until the root system has been established and the young wheat plant can obtain

§ Baking Technology 1,135 (1922).

proper food from the soil. In discussing some work of Kaker and Hulton †, who had carried on experiments to determine the value of the enzymatic constituents of flour, as they might affect bread making, Humphries has remarked that "these ferments were there to make the kernel of wheat available as food for the young plant," and that "Nature in making wheat, was more concerned with the reproduction of its kind than in feeding human beings."

It is not possible to give you any exact figures on the amount of gaseous chlorine necessary to add to any flour in order to produce that concentration of hydrogen ions so necessary to develop in the flour its highest baking value. I might perhaps say it is somewhere in the neighborhood of 150 parts per million. It may be more and it may be less. In a comparison of the different grades of flour we have to contend with a greater buffer effect in the lower grades than in the higher, so that the lower grades on this account require a larger amount of chlorine to produce the necessary intensity of acidity. This buffering effect is due probably to phosphates, and possibly to the proteids, too, as has been pointed out by Jessen-Henson, Luers and in others. Recent work of Bailey and Peterson ‡ lead them to the view that in the extracts from flours, the phosphates may be the principal buffers. They say that "electrometric titration curves show the extracts of high grade flours to be buffered less than the extracts of lower grades. It consequently requires less acid to bring the hydrogen ion concentration of a high grade or patent flour dough to the optimum for bread production than is required for a low grade flour. Possibly some of the difficulties experienced in making satisfactory bread from the lower grade or clear flours may be due to failure to obtain the optimum hydrogen ion concentration of the dough." They also say that "In view of the decided differences in the buffer action of high grade or patent, and low grade or clear flours, it is possible that some of the difficulties experienced in producing satisfactory yeast leavened bread from the lower grades may be due to failure to obtain the proper hydrogen ion concentration of the dough. Further experimentation with these lower grades of wheat flour, containing more than 0.60% of ash, should be conducted to ascertain the possibilities in this direction. Since conditions generally are unfavorable to the production of choice bread from clear flour, it is probable that a more careful adjustment, within narrower limits, is necessary than in case of doughs made from patent flours."

Within the limits of a paper of this character it is not practical to discuss the splendid experimental work which is being done on flours not only in this country but also abroad. The general trend of all these investi-

† Jour. Soc. Chem. Ind. 27,273 (1908).

‡ Jour. Ind. & Eng. Chem. 13,916 (1921).

gations is toward the utilization of physico-chemical methods in studying flour *per se*, as well as gluten. Out of all this work has come some splendid results and I hope that the time is not far distant when that most important scientific question of what constitutes the strength of flour will be solved. Whether the answer will be found revolving around the colloidal properties of the various flour constituents, remains to be seen. Already we know considerable about the effects of varying hydrogen ion concentration and of salts on the inhibition properties of gluten. This affects markedly the physical properties of the dough and the elasticity of the gluten. In order to get best baking results the gluten must have the proper elasticity and the dough the proper viscosity. The intensity of the acidity of the flour or dough is the most important variable in bread making. We know that when we control, in a proper fashion, the hydrogen ion concentration through the treating of flour with chlorine, we are on substantial scientific grounds. In this fashion, we are able to influence the viscosity of the dough until it becomes practically a minimum at which point the gluten seems to have the proper degree of elasticity. As Bailey and Peterson have said §: "Through the proper hydrogen ion concentration the elasticity of the gluten, and the activity of the yeast and of 'rope producing' bacteria are affected in the right direction."

Henderson, Fenn and Cohn* in their study of the "Influence of Electrolytes upon the Viscosity of Dough," remark that "* * * it is evident that with variation in hydrogen ion concentration, viscosity passes through a minimum a little on the acid side of pH5. Jessen-Henson found pH5 to be the best hydrogen ion concentration in bread making and it is also, as we have observed, the most favorable for the rising of dough." They also state that their studies indicate that so far as CO₂ production during fermentation is concerned, that the best results are obtained near the point of minimum viscosity.

It is also probable, that it is due to the favorable action of the hydrogen ion concentration on the activity of the yeast enzymes, that we find that the fermentation period of a dough is materially decreased, as we approach that point where the intensity of acidity is most favorable for the optimum baking results.

I think Cohn and Henderson† have summed up quite concisely, the value of acidity: "In the first place the desirable length of the fermentation is determined by the acidity of the dough. Increase in the acidity increases the activity of the yeast and shortens the fermentation. Consequently the amount of sugar required by the yeast is diminished (unpublished observa-

§ Jour. Ind. & Eng. Chem. 13,916 (1921).

* Jour. Gen. Phys. 1,387 (1919).

† Science 48, 50 (1918).

tions of the authors). Besides, the rate of carbon dioxide production of the yeast within the dough, the tenacity and elasticity of the dough, and the escape of gas from the dough, are dependent upon the acidity. Experience in this and other laboratories has shown that the best acidity for the baking of bread is indicated by the turning of methyl red from orange to red. *In sum, the acidity of the dough at the time of baking seems to be the most important variable factor in bread making.*"

I wish to quote once more, in this connection, from an article by Henderson ‡ who says: "It is possible, however, to measure the hydrogen ion concentration of bread by the addition of the ordinary solution of methyl red (0.02% in 60% alcohol) to the partly cut surface of the loaf. Three or four drops of the indicator should be placed upon a single spot and five minutes should be allowed to pass. Then, if the color is a full red without an orange nuance, the hydrogen ion concentration is approximately $10^{-5}N$, or more. If an orange tint develops, greater amounts of acid should be added to successive batches of dough until the test with bread just gives the desired color * * *."

So far as "rope" is concerned, I think it has been clearly established that the growth of the bacillus mesentericus which causes rope is practically inhibited at a pH of about 5.

I wish now to sum up what seems to me to be the main conclusion to be drawn from the foregoing discussion.

1. Any treatment of flour, to condition it for its best baking results, which does not increase the intensity of its acidity, does not condition the flour in the same manner as such flour is conditioned in storage. As Jessen-Hansen has so aptly put it: "Of the different artificial means advocated in recent times as being capable of ameliorating flour, none are of value other than those which increase the hydrogen ion concentration in the dough."

2. The proper control of the intensity of the acidity of flour, such as may be accomplished through treatment with chlorine, or any other means which accomplishes the same results, effects favorably those factors which are so essential to the baking efficiency of a flour.

3. The optimum intensity of acidity for the best results is such that methyl red should just turn red without any orange nuance, when this indicator is applied to the freshly cut surface of the oven product. This change in the color of the indicator is produced when the concentration of the hydrogen ions is about 10^{-5} normal, or, in other words, the pH is about 5.

4. The intensity of acidity is that factor which under proper control, favorably affects the dough viscosity, by reducing it towards its minimum

‡ Ibid 48,247 (1918).

and, at the same time, produces those changes which increases the elasticity of the gluten. It lessens the period of fermentation by increasing the activity of the yeast. It also acts favorably in the control of "rope."

5. Flour which has been subjected to the chlorine treatment, is stabilized in the sense that its improved baking capacity remains constant for many months. This stabilizing is brought about by arresting the actions of those enzymes to which is due the increase in acidity of an untreated flour when stored.

6. The acidity of flour is its most important variable. Hence the treating of flour with chlorine and the consequent production of the optimum intensity of acidity, is at once a great asset to the miller as it is to those who use his output and that means each and every one of us. This means of controlling this important factor—acidity—I consider a great advance in the milling art. Now, the flour buyer can obtain a stabilized flour at its enhanced baking capacity. Although not previously discussed, it may not be amiss to say that such chlorine treated flour is stabilized in yet another sense, and this is of importance, too. Such flour is much less readily subject to rancidity, mold and insect infestation.

Short Weights of Flour and Flour.

By W. L. FRANK,
Sherman Grain and Cotton Exchange, Sherman, Texas.

Recent activities of weights and measures inspectors in Texas followed by several hundred indictments charging a number of flour mills and cotton seed products manufacturers with short weights, has precipitated the milling industry of Texas into a legal battle that is being fought to a finish.

The Commissioner of Markets and Warehouses in Texas, is also ex-officio State Superintendent of Weights and Measures, and is charged with the administration of the Weights and Measures Laws. Deputy inspectors are employed who travel about over the State and check up the weights of package products as well as the scales and measures used by manufacturers and dealers.

Each sack of flour or other material, is weighed separately, and if found to be deficient in weight, the manufacturer is indicted and fined from \$15.00 to \$200.00. Each sack, whether from the same lot or shipment, constitutes a separate offense. No consideration is given to average net weights, and the fact that the excess weight of some sacks in a lot or shipment may compensate for the deficiency of others is disregarded.

The Texas law is similar to the Federal law, but it is not administered in the same way. From the language used, it appears that it was the intention to have the Texas law conform with the provisions of the Federal law.

The differences will be discussed under specific headings, namely: Specifications, Variations, Tolerances, and Exemptions.

LABELING.

One of the points raised in the controversy pertains to the label—or statement of weight. The defendant mills contending that the Federal law on this point is complied with when the sacks are marked “48 pounds when packed” and “24 pounds when packed.” The Texas Commissioner insists that the Texas statute requires them to be marked: “Forty-eight pounds net weight” and “Twenty four pounds net weight.” The reasonableness of the Texas Commissioner’s interpretation has been attacked on the ground that the weight of flour may vary after it is packed due to atmospheric conditions. This point will be brought out more clearly in connection with “Tolerances” and “Causes of Variations in the Weight of Flour.”

IS THE TEXAS WEIGHTS AND MEASURES LAW VALID?

The constitutionality of the Texas Weights and Measures Law was attacked in a “test case” tried in the District Court at Sweetwater, Texas, on April 13, 1922. The presiding judge refused to find the law unconstitutional. Attorneys for the defendant appealed the case to the Court of Criminal Appeals. The Higher court has not rendered its decision.

On April 24th last, a suit was filed by counsel for one of the defendant mills, asking an injunction to restrain the State Commissioner of Markets and Warehouses from further prosecution under the State Weights and Measures Law. “The law was attacked on several counts, there being eleven allegations that the act is unconstitutional.” (Quotation is from Dallas News, dated April 25, 1922.)

For the benefit of those who have not read a detailed account of the case, I will give the eleven allegations referred to above:

1. “That the acts and omissions denounced are not made offenses by the law.”
2. “That it is of doubtful construction and inoperative.”
3. “That so many laws are existing without repeal that it can not be said that the sale of a package without its true net weight being marked on it is a penal offense as defined in the written law.”
4. “That said section is too broad to be valid.”
5. “That it is unreasonable, unnecessary and arbitrary exercise of power as to deprive plaintiff of constitutional rights.”
6. “That it is unreasonable in that it prescribes an impossible thing and violative of the Federal Legislation.”
7. “That it is invalid in that it attempts to delegate legislative power to suspend a law to the Commissioner of Markets and Warehouses.”
8. “That it is void because its caption embraces two subjects.”

9. "That it is unconstitutional because it prescribes weights which only the Federal Congress can designate and had designated."

10. "That it imposes a burden on interstate commerce and is therefore void."

11. "That the law is not in effect because of rules for variations have not been fixed as contemplated in Chapters 130 and 131 of the Acts of the Thirty Sixth Legislature."

Each of the numerous allegations were argued at considerable length by the defense attorneys. While a detailed account of the arguments pro and con would perhaps be of interest to those charged with the making and enforcing of similar laws, and to those whose duty it is to see that their employers comply with the various state laws, space and time will not permit my going into detail.

Judge Cecil H. Smith, one of the defense counsel, in an editorial that appeared in the Dallas News dated May 6, 1922, made the following statement in reply to an editorial published in that paper a few days previous:

"The petitioning mill has a capacity of 2,000 barrels a day. If it should run at capacity for a day and pack one-half its output in forty-eight pound sacks and one-half in twenty-four pound sacks, usual sized, it would produce 12,500 sacks a day. If each sack should lose one per cent of its moisture or absorb one per cent of its moisture—the happening of either is a common occurrence—the mill would incur a liability on each sack; the agents of the department have pursued the course with this mill of preferring charges against three of the officers of the mill on each sack. For one day's run the mill would incur a minimum liability of \$1,875,000, or a maximum liability of \$8,437,500. For a year the minimum liability of this mill would be \$675,000,000 and the maximum \$3,037,500,000."

TEXAS WEIGHTS AND MEASURES LAWS.

There are three laws relating to Weights and Measures in Texas. The "Units of Weights and Measures Law" (H. B. No. 247), is given as:

"An Act to define what shall constitute a unit of weight or measure for all commodities purchased or sold by length, weight or measure * * *."

and Section 1 of the above specifies:

"SECTION 1. Legal Standards. The standard of weights and measures adopted and used by the government of the United States is hereby declared the legal standard of Weights and Measures of the State; * * *."

The "Public Weighers Law (H. B. No. 248)" described as: "An Act to define what constitutes a public weigher," is irrelevant to the subject—Short Weights in Flour. The third law designated as "General Weights and Measures Law (H. B. No. 249)" and described as:

"An Act to establish a standard of weights and measures in the State of Texas; * * *" is of special interest in that Section 5 of

that Act published as A 169-619-5M, page 22, and below the center of page, contains the following statement:

"* * * The State Superintendent of Weights and Measures shall establish tolerances and specifications for commercial weighing and measuring apparatus for use in the State of Texas similar to the tolerances and specifications recommended by the National Bureau of Standards, * * *

THE FEDERAL LAW.

The following quotations from Handbook Series of the Bureau of Standard No. 1, designated as a "Manual for Weights and Measures Officials," page 183 contains the following statement with reference to net weights:

"Net-Weight Amendment to the Food and Drugs Act.

37 Stat., ch. 117, p. 732, 62d. Cong."

"Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled, That section 8 of an Act entitled "An Act for preventing the manufacture, sale, or transportation of adulterated or misbranded or poisonous or deleterious foods, drugs, medicines, and liquors, and for regulating traffic therein, and for other purposes," approved June thirtieth, nineteen hundred and six, be, and the same is hereby, amended by striking out the words "Third. If in package form, and the contents are stated in terms of weight or measure, they are not plainly and correctly stated on the outside of the package," and inserting in lieu thereof the following:

"Third. If in package form, the quantity of the contents be not plainly and conspicuously marked on the outside of the package in terms of weight, measure, or numerical count: **Provided, however,** That reasonable variations shall be permitted, and tolerance and also exemptions as to small packages shall be established by rules and regulations made in accordance with the provisions of section three of this Act."

Continuing the quotation from the above mentioned publication, page 184:

"Regulation of Marketing the Quantity of Food in Package Form."

Under Section 3 of the Food and Drugs Act of June 30, 1906 (34 Stat. L., p. 768-772), as amended by the Act of March 3, 1913, entitled "An Act to amend Section 8 of an Act entitled 'An Act for preventing the manufacture, sale, or transportation of adulterated or misbranded or poisonous or deleterious foods, drugs, medicines, and liquors, and for regulating traffic therein, and for other purposes,' approved June thirtieth, nineteen hundred and six" (37 Stat. L., p. 732), regulation 29 of the Rules and Regulations or the Enforcement of the Food and Drugs Act has been amended by Food Inspection Decisions 154, 157, 163, and 168, so as to read as follows:

REGULATION 29.

Statement of Weight, Measure, or Count.

(Sec. 8, par. 3, under "Food," as amended by act of March 3, 1913).

(a) Except as otherwise provided by this regulation, the quantity of the contents, in all cases of food, if in package form, must be plainly and conspicuously marked, in terms of weight, measure, or numerical count, on the outside of the covering or container usually delivered to consumers."

Continuing the quotation from the bottom of page 185 and 186:

"(h) The quantity of the contents may be stated in terms of minimum weight, minimum measure, or minimum count, for example, "minimum weight 10 oz.," "minimum volume 1 gallon," or "not less than 4 oz.;" but in such case the statement must approximate the actual quantity and there shall be no tolerance below the stated minimum.

(i) The following tolerances and variations from the quantity of the contents marked on the package shall be allowed:

(1) Discrepancies due exclusively to errors in weighing, measuring, or counting which occur in packing conducted in compliance with good commercial practice.

(2) Discrepancies due exclusively to differences in the capacity of bottles and similar containers resulting solely from unavoidable difficulties in manufacturing such bottles or containers so as to be of uniform capacity: Provided, That no greater tolerance shall be allowed in case of bottles or similar containers which, because of their design, can not be made of approximate uniform capacity than is allowed in case of bottles or similar containers which can be manufactured so as to be of approximate uniform capacity.

(3) Discrepancies in weight or measure, due exclusively to differences in atmospheric conditions in various places, and which unavoidably result from the ordinary and customary exposure of the packages to evaporation or to the absorption of water.

Discrepancies under classes (1) and (2) of this paragraph shall be as often above as below the marked quantity. The reasonableness of discrepancies under class (3) of this paragraph will be determined on the facts in each case."

The variation in weight of package foods has been thoroughly investigated by the United States Department of Agriculture, Bureau of Chemistry. A bulletin (13) has been published on the subject, from which the following quotation was selected as indicating the position which that Bureau has taken on the subject:

"The food official and the food packer often ask, "What is the variation in weight of package foods?" * * * It is recognized that an answer might be obtained by different methods, but the steps by which an answer has been reached in this bulletin are (1) selecting specifications for a method of weighing to represent good commercial practice in packing granular, free-flowing products, (2) calculating the maximum errors expected from these specifications, and (3) showing that the calculated errors approximate the variation in weight found by the commercial application of the specified method."

The unreasonableness of the interpretation under the Texas Laws relating to Weights and Measures, was stressed by the defense counsel in their pleas to the courts in both the *habeas corpus* and the injunction suits. It was contended that the specifications, variations, tolerances and exemptions established by the Federal Government relative to the weight of the packaged foods, should be recognized by the Commissioner of the Department of Markets and Warehouses of Texas. It was alleged that the State of Texas had discriminated against Texas millers by exacting more rigid specifications, variations, tolerances and exemptions on products manufactured in Texas, than those imposed by the Federal Government on products shipped in Interstate Commerce. †

The question, "What Constitutes Flour?" is perhaps as important as is the matter of weight. Both Federal and State Standards will be quoted, also a portion of the Texas law. The Federal definition of flour, is:

"3. Flour is the fine, clean, sound product made by bolting wheat meal, and contains not more than thirteen and one-half percent (13.5%) of moisture, not less than one and twenty-five hundredths per cent (1.25%) of nitrogen, not more than one per cent (1%) of ash, and not more than fifty hundredths per cent (0.50%) of fiber."

The Texas definition of flour, is:

"3. Flour is the fine, clean, sound product made by bolting wheat meal and contains not more than thirteen and one-half percent of moisture, not less than one and twenty-five hundredths percent of nitrogen, not more than one percent of ash, and not more than fifty hundredths percent of fiber."

The Texas Law reads in part as follows:

"* * * It shall also be the duty of the Dairy and Food Commissioner to formulate, publish and enforce such rules and regulations as may be necessary to enforce this Act, and he shall adopt the standards for foods, food products, beverages, drugs, etc., and the methods of analysis authorized as official by the United States Department of Agriculture in so far as they are applicable in the light of modern discovery and scientific research."

The intention of the legislature and the import of the Texas Pure Food Law and the standards for foods, etc., adopted or to be adopted in Texas, are stated in terms so plain and clear as to leave no room for doubt.

Evidence produced at one of the trials, previously mentioned, was to the effect that the defendant made flour containing 13.50% moisture, and it was contended that flour ordinarily did not retain that amount of

† Note:—This point will be considered more in detail under the third Section of this paper, entitled "Specifications, Variations, Tolerances, and Exemptions."

moisture when stored for any great length of time. The defendant further contended that the difference in moisture content in flour as made and after storage was a matter not within his power to control, and that this factor of variable moisture content, could affect the net weight of a sack of flour.

CAUSES OF SHORT WEIGHTS.

There are a number of things that may cause short weights in the net weight of packaged products. The worst of these is fraud. Whether it be by the use of inaccurate scales or weights, or by deliberate design on the part of the individual operator, is immaterial. However, it was not proven in the cases mentioned, that fraud had been practiced. The indictments against the defendants were based on the weights as found by the inspector when he weighed the flour and other materials.

Another frequent cause of variations in the net weight of flour is that of moisture content. This factor may vary from day to day, so that a 48 pound sack may gain or lose as much as two or three ounces in 24 to 36 hours, and greater amounts in longer periods. The subject has been studied by several investigators according to Bailey,[†] who mentions Brewer, this paper.

Willard, Neumann, Guthrie and Norris, Sanderson, Swanson, Willard and Fitz, and Stockham. The writer has not had an opportunity to read three of these papers mentioned.

The importance of moisture content of flour was recognized by Bailey, whose statement on the subject is quoted herewith:

"Significance of Moisture Content of Flour."

It seemed desirable to ascertain the moisture content of flour in atmospheres of differing but constant humidity, after a period of exposure sufficiently long to permit the hygroscopic moisture of the flour to reach equilibrium with the atmosphere. Such data would be of service in a number of ways. Shippers, purchasers, and food control officials dealing with flour need more precise information concerning changes in moisture content, and consequently in the net weight of flour packages. The baker and store keeper need such data for the same reason, and, in addition, are concerned with the indirect effect of changing moisture content upon the keeping qualities of flour on prolonged storage. Flour which reaches a high moisture content is quite likely, if kept moderately warm, to become unsound through the activity either of its own enzymes, or those of fungi, and especially molds and related forms, which develop on the moist flour. Millers may find such data of service in developing methods of controlling the atmospheric humidity in mills and certain milling machines."

Bailey's Conclusions are pertinent to the subject at issue in Texas, and are quoted herewith:

[†] Note:—Reference is to literature cited. See list of references at end of

Conclusions.

Flour responds readily to changes in the humidity of surrounding air, the rate at which equilibrium in moisture content is approached depending apparently upon conditions of exposure.

Hygroscopic moisture in flour in equilibrium with atmospheric humidity at 25 degrees C. ranges from a little more than 5 per cent of moisture at 8 degrees per cent relative humidity.

Curves representing the relation between hygroscopic moisture (ordinates) and relative humidity (abscissae) have the shape of a simple parabola, thus indicating that hygroscopic moisture does not increase at a uniform rate when in equilibrium with an increasing atmospheric humidity.

The following quotation from the same author is of special interest as indicating the rate of changes in weights:

"The question at once arises as to how rapidly these flours change in moisture content with variations in the humidity of air to which they are exposed. That the response is rapid may be deducted from the experiments of Guthrie and Norris (1912). The exact rate must depend upon a number of variables, however, including the size and shape of the package in which flour is contained, the material from which it is manufactured, the extent of circulation of air about the package, and possibly other factors of minor importance. * * * Such data as were secured in certain of our preliminary studies indicate, as might be anticipated, that a dry flour placed in a humid atmosphere (or vice versa) changes rapidly the first three days, and much more slowly the next three days, after which, if exposed in thin layers, there is little further change.

If we assume that atmospheric moisture affects the hygroscopic moisture content of that flour in a sack within one inch of the outer surface, we may compute the necessary change in moisture content to effect a change in weight of one ounce. For the purpose at hand we may consider a 48 pound sack of flour to be represented by a cylinder 27 inches high and approximately 30 inches in circumference. A layer of flour one inch in thickness in such a sack would represent approximately 500 cubic inches of space, or about one fourth of the cubical contents of the sack. A forty-eight pound sack should contain 768 ounces of flour. One fourth of 768 equals 192 ounces. One ounce is 0.52 per cent of 192 ounces. Thus we see that a change in the moisture content of a layer of flour one inch deep all the way around a sack of flour of only a trifle over one-half of one per cent, would result in a change in net weight of one ounce. A two per cent change in moisture content would produce a 4 ounce variation in weight, and a five per cent change in moisture content would result in a 10 ounce variation in weight.

Dr. S. J. Crumbine, Chief Food Inspector, and Secretary of the Kansas State Board of Health, suggested to the Kansas State Agricultural College and Experiment Station, that tests be made on flour to determine the extent

of loss in weight during storage. The tests were made and published in a bulletin (17). The following quotations are from the above mentioned publication, beginning at bottom of page 119:

"Record of the Changes in the Weight of Stored Flour.

The complete record of the weight of these sacks taken at different times during the period of a year is found in table XXVI. In the first column are the weights obtained as soon as the flour arrived in the laboratory. This includes the weight of the sack itself, together with the flour. The average weight of empty flour sacks is about one-fourth of a pound. Most of the sacks were full weight, and considering the conditions under which flour must be weighed in a mill, the weights were fairly uniform. The results of subsequent weighings are shown in the other columns. The greatest loss occurred during the first two months, and the flour continued to lose throughout the winter.

TABLE XXVI.
Changes in the Weight of Stored Flour—Pounds.

Sack	Aug. 11	Sep. 11	Oct. 11	Nov. 17	Jan 11	Mar. 11	Apr. 11	May 13	June 14	Aug. 10
1	48¼	48¼	48	48	47¾	47½	47½	47½	47½	47¾
2	48¼	48	47½	47¾	47½	47¾	47¼	47¼	47¾	47½
3	48¼	48	47½	47¾	47½	47¾	47¼	47¼	47¾	47½
4	48	48	47¾	47¾	47¾	47½	47¾	47¾	47½	47½
5	48	48½	48½	47¾	47¾	47¾	47¾	47½	47½	47¾
6	48	47½	47½	47¾	47½	47½	47¾	47¾	47½	47½
7	48¼	48¼	48	47½	48	47½	47½	47½	47½	47½
8	48¼	48	48	47¾	47¾	47¾	47½	47½	47½	47¾
9	48¼	48½	47½	47¾	47¾	47½	47¾	47¾	47½	47¾
10	48¼	48½	48	47¾	47½	47½	47½	47½	47½	47¾
11	48¼	48	47½	47¾	47½	47¾	47¼	47¼	47¼	47¾
12	48¼	48	47½	47½	47½	47¾	47¼	47¼	47¾	47½
13	48¼	48½	48	47½	47½	47¾	47½	47½	47½	47¾
14	48¼	48	47½	47¾	47½	47½	47½	47½	47¾	47½
15	48¼	48	47½	47¾	47½	47½	47¾	47¾	47½	47½
16	48¼	48¼	48	47¾	47½	47¾	47½	47½	47¾	47½
17	48¼	48½	48	47½	47¾	47¾	47½	47½	47¾	47¾
18	48¼	48½	48	47¾	47¾	47½	47¾	47½	47¾	47½
19	48¼	48½	47½	47½	47½	47½	47¾	47¾	47½	47½
20	48	47½	47¾	47½	47¼	47½	47¼	47¼	47¼	47½
21	48¼	47½	47½	47½	47¾	47¼	47¼	47¼	47¾	47½
22	48½	48	47¾	47½	48¾	47½	47½	47½	47½	47¾
23	48¼	48	47½	47½	47½	47½	47¾	47½	47½	47½
24	48¼	48	47½	47¾	47½	47½	47¾	47¾	47½	47¾
25	48	48	47¾	47½	47½	47½	47¾	47½	47¾	47¾
26	48½	48	47½	47½	47½	47½	47¾	47½	47½	47¾
27	48½	48	47½	47½	47½	47½	47¼	47¼	47½	
Av.	48.19	48.05	47.90	47.73	47.65	47.55	47.40	47.43	47.55	47.67

The lowest average weight for all of the sacks was in April, when the average loss had amounted to a little more than three-fourths of a pound per sack. A very large number of the sacks had lost a pound in weight. After April there was a slight increase in the weight of some of the sacks. This was sufficient to raise the average weight a small amount. However, this increase in weight continued through the summer months. None of the sacks regained their original weight. It seems, therefore, that when this flour was fresh it contained more moisture than it would retain under such hygroscopic

conditions as were present in this store room. It is reasonable to conclude from this experiment that a 48 pound sack of flour will lose three-fourths of a pound or more in weight if stored in a light, steam-heated room. The moisture content of this flour was 10.58 per cent. This is over one per cent lower than the average moisture content of the commercial flours reported in the preceding pages. If these sacks lost nearly a pound under the storage conditions present, it is reasonable to expect that flour with a higher moisture content will lose more. This loss will be governed by two factors: the hygroscopic conditions of the air and the moisture content of the flour. The drier the air and the more moisture the flour contains, the greater will be the loss. Reverse the conditions and the loss will be small, or there may be an actual gain in the weight.

Continuing the quotation from the middle of page 126,

"Second Part."

Tests on Unbleached Flours, Different Conditions of Storage.

To test the effects of different conditions of storage on flour, a straight unbleached flour was selected. This was of the same grade as the unbleached flour used in the preceding work. There were three storage conditions: The attic of a stone building, the steam-heated room, and sealed cans. The first represents the condition of the unheated warehouse; the second the same conditions as were used for the comparison of the bleached and unbleached flours and would be the condition of the ordinary store; the third gives the conditions of a warm room, but without a chance for any change in moisture content. * * *

"* * * The season was alternately very wet and very dry, and the result shows that flour is very sensitive to moisture changes in the atmosphere. In the steam-heated room there was a constant loss in the fall and winter, with the maximum loss in April when the steam heat ceased to be used. After this there was certain gain.

These results show that the weight of flour varies with atmospheric conditions. Two per cent may easily be lost when flour is stored in such a room as an attic, or any place which gets very warm in the late summer. Flour will partially regain weight when the atmosphere becomes more humid. * * *

Continuing the quotation from the bottom of page 127,

"Second Season's Storage Experiments—1910-'11.

Condition and Arrangement of Samples.

For the work on stored flour the second season the same storage conditions as the previous year were used; namely, the unheated room, a steam-heated room, and sealed jars. * * *

The sacks stored in the unheated room showed a loss of nearly half a per cent the first thirty days. The moisture content of the flour samples at the beginning was nearly thirteen per cent. The loss, though small, continued during the fall. This loss was partially regained during the winter. A comparatively large loss occurred during the spring months, after which the weight was constant. The season was dry in the fall, wet in February, and dry in the spring

and summer. * * *

In the steam-heated room the loss was less than in the attic room during the early fall. The samples stored in the steam-heated room lost most in the winter. This loss was partially regained in the spring and summer.

Table Showing Pounds of Loss Per One Hundred Pounds of Flour at the different dates.

Date of Weighing	Aug 10 1910	Sep 10 1910	Oct 10 1910	Dec 10 1910	Feb 10 1911	Apr 10 1911	Jun 10 1911	Aug 10 1911
In unheated room		.47	.55	.70	.48	1.00	2.62	2.62
In steam-heated room, not bleached		.24	.26	1.91	2.40	2.94	2.28	2.91
In steam-heated room, ordinary bleached		.22	.27	1.90	2.54	2.90	2.31	1.81
In steam-heated room, strongly bleached		.17	.21	1.85	2.52	2.97	2.36	1.84

The above table shows the number of pounds of probable loss in one hundred pounds flour stored under the conditions named. It should be noted that where flour is stored in larger quantities the loss will likely be less. However, as was shown in the experiments with the forty-eight pound sacks, the loss may be as much as two per cent in flour that was lower in moisture content than the average. A loss of two and one-half per cent is not improbable even from the large sacks.

Chemical Composition of Flours Stored Under Different Conditions.

TABLE XXXIII.

The moisture was determined by drying in a vacuum desiccator until constant weights were obtained. There was a decrease of nearly one per cent of moisture in the flour stored in the sealed cans. The decrease of moisture is not regular throughout the year. It was noticed that as the samples became older, longer and longer time was necessary to obtain constant weights. Some of the older samples were kept in the desiccator for three weeks and a continued slight decrease was noticed. It seems that there is a tendency for a part of the hygroscopic water to combine in some way with the flour so that it is less easily driven off. Other things being equal, it is not possible to obtain as large a per cent of moisture from old flour as from new. These cans had not lost any weight, yet there was a decrease of about one per cent of moisture. This had combined with the flour so that it could not be driven off by the methods used in drying. * * *

There are several points brought out in the work of Swanson, Willard and Fitz which ought to be considered as affecting the subject—moisture content and short weight in flour. I will enumerate them:

1. The weight of a sack of flour varies.
2. Flour may gain or lose moisture during storage.
3. Relative humidity of the air and the moisture content of the flour govern the loss in weight that may occur.

4. Flour is sensitive to atmospheric moisture.
5. As much as two per cent of its weight may be lost by a sack of flour in storage, and in some cases more.
6. The moisture content of flour in air tight vessels decreases with age, although the weight may remain the same.

In the Kansas report, the original moisture content was given as 10.58 per cent, or approximately three per cent less than the standard specifications. The Kansas flours which contained only 10.58 per cent moisture, lost nearly two per cent. Had the Kansas flour contained 13.50 per cent moisture the loss would have been close to five per cent, that is, between 2.0 and 2.5 pounds per forty-eight pound sack.

Stockham (16) studies on "The Capacity of Wheat and its Products for Atmospheric Moisture Under Saturated and Dry Conditions," should be mentioned as he has conducted rather extensive investigations on the subject:

"The limiting capacity of wheat and its products diminishes with increase in temperature. At 100 degrees C its capacity is nothing with ordinary conditions of humidity. It increases with a decreasing temperature and reaches a maximum at 0 degree C. Above 60 degrees C it is dependent largely upon chemical changes which in turn are dependent upon the relative amount of moisture present and available. With the humidity zero, the capacity is zero regardless of the temperature, and at the saturation point at any temperature the theoretical limit to the capacity is the same as the water absorption at that temperature. In the saturated atmosphere, however, the theoretical limit is rarely or never more than half reached by wheat or its products at temperatures, 0 degree to 40 degrees C because of the secondary changes, the influence of which depend upon the foreign spores and bacteria present, temperature, and proportion of water. * * *

Another interesting observation recorded by Stockham (16) is an increase in the water absorbing power of flour during storage. Eight months after storage, the average water absorbing power of 34 samples of flour increased over one per cent, and the moisture absorbing power of one sample increased 2.9 per cent during a 15 month period.

Tabulations are given, showing the moisture content of flour under different conditions of storage. Two tables are reproduced herewith as showing something of the rapidity of the changes in moisture content of flour.

Composite Sample Flour, (7) Saturated Atmosphere

Time Exposed		Moisture
Days	Hours	Per cent
0	0	11.97
0	4.66	14.77
1	23.08	22.86

5	3.5	26.87
6	22.17	27.50*
9	2.92	28.74
11	3.42	25.10

* Mould growth general and visible to the naked eye.

Composite Sample Flour, (10) Dry Atmosphere		
Time Exposed		Moisture
Days	Hours	Per cent
0		11.97
1		1.52
2		.90
4		.27
7		0.00

Sanderson (14) made "A Study of the Variation in Weight of a Fifty Pound Sack of Flour During Storage." In commenting on the experiment, Sanderson wrote:

"The enforcement of the Net Weight Law of the State of North Dakota suggested the experiment, * * *"

Thirty-eight samples were used in the experiment, each having an original moisture content of 9.86 to 12.42 per cent. The occasion for running the experiment, the original moisture content of the flour used, and the conclusion drawn, are pertinent to the points at issue in Texas. The conclusions are in part as follows:

"The variation in weight is due largely to atmospheric conditions and is influenced by the same, because during the spring and early summer moisture was lacking and ran below normal and during that period was when the greatest loss in weight was experienced. Then during the latter part of June and July when the rains came all samples show material gains. That temperature and condition of store room and atmospheric conditions should be considered when enforcing the food law.

That flour is more susceptible to variations in the summer months than in the winter.

Wheat milled with a high temperature content is more liable to produce a flour that will lose in weight in storage than a wheat of low moisture content.

That moisture added in the tempering of wheat is, in this climate, very largely lost again in the storage of the flour."

In a later publication, Sanderson refers to his previous work on weight of flour in storage as follows:

"A study of the data of the above experiment indicates that to base any hard and fast rule on the same for our government in the future would be wrong, because of the fact that we had some sacks

of flour that gained in weight while others lost during the same period of time and under what we believed to be the same conditions of storage. Also we had flour manufactured from what we believed a homogeneous sample of wheat to show almost as wide variation in weight stored in the same pile as flour made from the different samples of wheat. We refrained from drawing any definite conclusions and planned to make a further study of the problem."

"A second experiment was undertaken in which twenty samples of flour were used. At the close of the second experiment, Sanderson drew the following conclusions:

"In drawing conclusions from these two tests there is a chance for a difference of opinion on some of the points here spoken of and we will be surprised if we are not criticised on some of them. However, we are making them in a spirit of fairness as we see it and in many of them we have the results shown on which to base our conclusions. For instance, in the matter of moisture in flour. The allowable moisture content in interstate commerce is 13.5 per cent and in localities having a humid atmosphere might be considered the natural normal moisture while the same flour shipped into a locality with an arid atmosphere might show a moisture content of between eight and nine per cent. Therefore, for the benefit of the consumer and to place no hardship on the manufacturer in any locality we say the standard moisture content should be placed at about 11 per cent and then a reasonable amount of variation would protect the honest manufacturer and consumer alike and make the law effective as it was intended. * * *

Conclusions.

The flour with about 11.0 per cent moisture will vary less in weight than if the moisture be more or less.

That the normal moisture content of flour in this climate seems to be about 11.0 per cent.

That the normal moisture content of wheat in this climate seems to be about 13.0 per cent.

That flour made from wheat with normal moisture 13.0 per cent with not to exceed 2.0 per cent added moisture in tempering will produce a flour with moisture content of about 11.0 per cent that will not vary in storage more than 2.0 per cent and the variation is as liable to be above as below the original weight, owing to atmospheric conditions.

That ordinarily wheat with moisture content of between 14 and 15 per cent moisture is in prime condition to mill."

The position Sanderson took on the moisture content of flour is of interest in that it suggests a modification of the Federal Standard which allows 13.5 per cent moisture, and which would in substance specify the amount of *dry matter*. The advantage of using the weight of dry matter, as the basis of determining short weight in flour is obvious. The amount of dry matter remains practically uniform, if we disregard the small loss

from "dusting" through the sack, and these chemical changes alluded to by Swanson (17) which result in a decrease in moisture content without a decrease in weight of the sample. The following quotation from Sanderson will make his position clear:

"It has been proposed that the standard for moisture in flour be fixed at 11 per cent and that instead of limiting the per cent of moisture to 11 per cent, the standard shall call for a flour to weigh 49 pounds per sack, or 196 pounds per barrel, when calculated to 11 per cent basis for moisture. This would be entirely fair to all parts of the country.

At the present time there seems to be a tendency on the part of millers to introduce sufficient moisture to bring the per cent of the limit as fixed by the standards. If, on the other hand, we place the limit at 11 per cent we may do injustice to some parts of the country, but if the standard be fixed at 11 per cent as the basis for making the weight, there would be no inducement for the millers to add more water than is necessary to secure the best results for milling, and yet, no injustice would be done to those who might mill wheat that would produce a flour containing more than 11 per cent, providing, they furnished the necessary amount of flour to make up the difference."

Jago (12) gives some interesting data on the "Effect of Humidity of Air Moisture of Flour." He also gives the moisture content, and computed the highest and lowest weight of five 100 pound sacks of flour during a 16 day period. The daily weight of one sack of flour and the relative humidity of the air each day are included.

"630. Effect of Humidity of Air on Moisture of Flour—Flour is exceedingly hygroscopic and absorbs or loses moisture, according to whether the atmosphere is damp or dry, with great readiness. Richardson examined a series of flours immediately on coming from the mill, and again after being exposed to the atmosphere for a day, with the following results:

	Original Moisture	Gain or Loss	Second Day
No. 1	9.48	- -0.65	10.13
No. 2	7.80	- -2.15	9.95
No. 3	7.85	- -2.30	10.15
No. 4	7.97	- -2.15	10.12
No. 5	13.69	- -3.28	10.41

It will be seen that, notwithstanding the wide differences in percentage of moisture on the first day, they had, at the end of the second, become practically equalized. Richardson next allowed these flours to remain exposed to the atmosphere for 16 days, making during that period 15 determinations of moisture. In one and the same flour during that time variations of nearly 5 per cent were observed. In the following table the results are expressed in weights in pounds, which 100 pounds of the original flour would have assumed under the conditions:

	Original Weight	Original Moisture	Highest weight during 16 days	Lowest weight during 16 days	Amount of Variation
No. 1	100 pounds	9.48	102.88 pounds	99.53 pounds	3.35 pounds
No. 2	100 pounds	7.80	104.87 pounds	100.00 pounds	4.87 pounds
No. 3	100 pounds	7.85	105.20 pounds	100.00 pounds	5.20 pounds
No. 4	100 pounds	7.97	105.95 pounds	100.00 pounds	5.95 pounds
No. 5	100 pounds	13.69	100.00 pounds	95.35 pounds	4.65 pounds

No. 1 of these flours was the well-known brand, Pillsbury's Best; it will be of interest to give the weight of this each time determined, and also the relative humidity of the air each day.

Date	Weight of flour	Relative hu- midity of air
March 7	100.00 pounds	
March 8	100.65 pounds	46.4
March 10	99.53 pounds	35.0
March 11	101.73 pounds	59.0
March 12	102.68 pounds	60.1
March 13	99.88 pounds	34.0
March 14	101.08 pounds	
March 15	101.53 pounds	48.2
March 17	100.38 pounds	42.2
March 18	101.88 pounds	59.5
March 19	102.03 pounds	60.1
March 20	102.48 pounds	55.6
March 21	101.43 pounds	51.8
March 22	101.68 pounds	51.1
March 24	102.88 pounds	66.9

It will be observed that with an increased dampness of the air, the weight of the flour is also increased. Of course in strictness, the weight of the flour is governed by the degree of humidity prior to the moisture determination, rather than that at the time the determination is actually made.

On exposing a sample of patent flour to an atmosphere kept absolutely saturated with water, it absorbed more than 26 per cent of its original weight in 64 hours. The following table gives the weight at different intervals:

Weight of flour taken	1.0000 grams
Weight after 35 minutes	1.0285 grams
Weight after 18 hours	1.0930 grams
Weight after 22 hours	1.2005 grams
Weight after 42 hours	1.2405 grams
Weight after 64 hours	1.2670 grams

These variations in weight of which flour is capable go far toward explaining discrepancies in water-absorbing power, and yield, of laboratory samples."

Herman and Hall have determined the moisture content of a sack of flour on 30 consecutive days. Temperature and relative humidity were determined daily at time analysis samples were taken. In 18 days the

lowest moisture content was reached. During that period the moisture content decreased 2.85 per cent. In commenting on the subject, "Variation in Moisture Content of Flour During Storage," the authors made the following statements:

"The moisture content of flour is continually changing during storage and transit due to variations in climatic conditions. These fluctuations vary in severeness of course, depending upon the season of the year and the locality. In this relation relative humidity has the greatest influence. It should be remembered that water vapor is always present in the air due to the constant evaporation taking place, and the percentage present is dependent upon the temperature, as warm air will hold more vapor than cold air. Flour stored under conditions of high relative humidity will absorb moisture from the air, while a low relative humidity (the temperature remaining constant) will cause evaporation. Flour milled in the Southwest will usually lose at least one-half of one per cent of moisture during the first week, but on long hauls to eastern or southern points there is quite often a gain."

The writer, (6) in an unpublished report, gives the results of an experiment to determine the effect of atmospheric moisture upon the weight of flour. Four brands each of hard and of soft winter wheat flour were used. Three samples were taken from each brand. One was placed in a closed vessel over concentrated sulphuric acid, the second sample was placed in a closed vessel over distilled water, and the third was used in determining the moisture content of the original sample. The summary covering the last mentioned experiment is given herewith: (6)

"SUMMARY

1. Flour loses weight due to reduction of moisture content, when exposed to a dry atmosphere.
2. Flour gains weight due to increased moisture content, when exposed to a moist atmosphere.
3. Flour has a strong affinity for hygroscopic moisture as was indicated by the increase in weight of moisture-free flour, when placed in a closed glass vessel containing concentrated sulphuric acid. (The acid contained a small though undetermined amount of water.)
4. There was no appreciable difference observed in the percentage rate of increase in the weight of moisture-free hard and soft winter wheat flours.
5. Hard winter wheat flours of normal moisture content, (12.00—14.00%) retained more moisture while confined in a dry atmosphere for a given length of time, than did soft winter wheat flour, and absorbed a relatively greater amount when confined in a moist or humid atmosphere.
6. The original moisture content of flour (when milled) does not

materially affect its capacity for holding hygroscopic moisture under the conditions that obtained in the experiments described.

7. The moisture content of flour is influenced by the relative humidity of the adjacent atmosphere. This phenomenon can be described as an equilibrium between the moisture content of flour and of the surrounding air. The moisture holding capacity of flour increases with a higher relative humidity of the air, temperature remaining constant."

It has been said that the miller can not control the moisture content of the flour after it is made and stored under varying conditions of weather, etc. It is also true that the average miller is unable to control the moisture content of his flour at the time of manufacture except within rather wide limits. The writer (5) found that the difference in moisture content of flour and wheat varied on different days, the flour ranging from 0.55 per cent to 1.65 per cent lower in moisture content than the wheat after tempering. The following observations are quoted (5):

"1. Moisture content of flour was relatively lower on clear, warm days, * * *

3. Moisture content of flour was relatively higher on cloudy days and on days when it rained."

The conclusions drawn in connection with the above observations are in part as follows:

"3. Temperature, precipitation and general weather conditions may affect the relative moisture content of the flour as compared with that of the wheat from which it was made.

4. Relative humidity at times is a factor, as for instance a marked drop in temperature, or a day following a rain is apt to be accompanied by a relatively higher moisture content in the flour."

The invisible loss in milling has been considered by Bailey who says in part:

"The total loss incurred in milling cleaned wheat may be classified under two heads, (1) the visible or mechanical loss * * * and (2) the invisible loss resulting from evaporation of moisture.

* * * The principal factors * * * were:

- (1) Length of system, * * *
- (2) Temperature and humidity of the air, * * *
- (3) Temperature of the rolls, * * *
- (4) Ease of reduction of the middlings, * * *
- (5) Quantity of water employed in tempering, * * *
- (6) Moisture content of the wheat before tempering, * * *

On April 26, 1922, the writer begun an experiment to determine the effect of temperature and relative humidity upon moisture content and weight of flour and mill feeds. Duplicate sacks of bran (100 pounds), shorts (100 pounds) and flour (48, 24 and 12 pounds) were placed in an ordinary store room. One sack of each pair is weighed daily, and a sample

taken for moisture test from the other sack. A continuous record is being kept of both temperature and relative humidity. The experiment is still in progress. The following facts have been disclosed to date:

1. The gross weight of a 100 pound sack of shorts has decreased from 100 pounds, 6 ounces, on April 26, to 97 pounds, 6 ounces, on June 1. The moisture content has decreased in the same period from 13.77 per cent to 12.12 per cent.

2. In the case of bran, the weight decreased from 100 pounds, 4 ounces, to 97 pounds, 5 ounces, and the moisture content from 14.58 per cent to 11.93 per cent.

3. As there were three sizes of flour sacks employed in the experiment the data will be given in tabular form:

Size of sack, pounds	48	24	12
Original weight	48-2	24-2	12-2
Weight June 1	47-2	23-8	11-12
Loss in weight, ounces	16	10	6
Original moisture content	13.63	13.66	13.29
Moisture content June 1	12.21	12.23	12.00

4. The relative humidity averaged nearly 70 per cent during the first five days of the experiment. The first week of May the average relative humidity was about 65 per cent. For the second week in May, about 60 per cent, and for the third and fourth weeks 55 and 50 per cent respectively.

5. The temperature readings are less variable. Averaging about 23 degrees C during the first five days and for the first, second, third and fourth weeks in May, approximately 27, 28, 28 and 31 C degrees respectively. Large daily variations occurred in temperature.

Notwithstanding the high relative humidity during the first five weeks of the experiment, both the shorts and bran lost weight and decreased in moisture content. The same is true in the case of flour but to a less extent. At times the weight of both flour and feed remained stationary for several days or even showed small gains, after which it would lose weight.

In computing the loss in weight of flour, it is incorrect to add the percent difference in moisture content and assume that the result is the original weight. Duvel (3) has shown the fallacy of such a method in considering the "Moisture Content in Shrinkage in Grain." The following quotation from Duvel (3) will illustrate the point in mind:

"* * * the reduction in the percentage of moisture is not the same as the percentage loss in weight, and should not be the same if the moisture content is properly determined and the weighing

done correctly, notwithstanding the fact that water is the only substance liberated in the drying process.

Why the Reduction of Moisture and the Shrinkage in Grain are not the Same.

The difference between the reduction in the percentage of moisture and the shrinkage exists because there is a constant change of base in making the moisture test. Moisture percentages are calculated on the basis of the wet sample, * * *

To show how percentage decrease in weight differs from percentage decrease in moisture, the following example is given: Flour which originally contained 13.50 per cent moisture is dried down to 12.00 per cent moisture. Assuming that the original weight of the lot of flour contained 13.50 per cent moisture weighed 100 pounds to find the weight of flour in same sack containing 12.00 per cent moisture. Originally the sack contained 86.50 pounds dry matter. Reducing the moisture content from 13.50 to 12.00 per cent does not affect the weight of dry matter, but does change the per cent of dry matter. To illustrate:

$$86.5 \text{ pounds} = (100 - 13) = 87$$

$$87 \text{ pounds} = 87\% \text{ of what number?}$$

$$(87 \div .87) \times 100 = 98.97 \text{ pounds.}$$

Thus we see that the weight of the flour after drying is 98.97 pounds although the loss in moisture was only 1.50 per cent. Adding the per cent decrease in moisture (1.50 per cent) to the actual weight of the flour, (98.97 pounds) the sum would be 99.97 pounds.

In as far as the accessible literature has permitted, the writer has studied the work of previous investigators on moisture content of flour. In several instances additional deductions have been made by the writer showing the application to the Texas situation of the principles and facts developed by previous investigators. The facts disclosed may be summarized as follows:

SUMMARY.

1. The moisture content of flour varies.
2. The extent of the variations depends largely upon three things, namely, (a) conditions of exposure, (b) relative humidity, and (c) original moisture content.
3. Variations in the weight of flour result from changes in moisture content.
4. Per cent decrease in moisture content of flour is not equivalent to the corresponding per cent decrease in weight.
5. The original moisture content of flour at time of manufacturing cannot be controlled except within rather wide limits.
6. The moisture content of flour during storage cannot be controlled by the manufacturer.

There are other causes of variations in the weight of flour. The

writer has considered several of these in an unpublished report (7). The location of ceiling fans directly above flour packing scales, and the effect of drafts and currents through the flour packer's room upon the balance of scales have been mentioned as possible sources of error in the weight of flour.

Other sources of errors in the weights of flour are the tolerances allowed in the weights and scales used in the weighing operations. These will be considered in detail under the heading "Specifications, Variations, Tolerances and Exemptions."

PART III.

SPECIFICATIONS, VARIATIONS, TOLERANCES AND EXEMPTIONS

Applicable to the Weight of Flour, Etc., Under the Food and Drug Act of the United States.

SPECIFICATIONS.

Composition.

Moisture being the only composition factor involved in the Texas controversy, only a portion of the Federal definition of flour will be quoted: (18)

"* * * and contains not more than thirteen and one-half per cent (13.50%) of moisture, * * *"

Quantity or Weight.

The Federal regulations as to quantity or weight are specific, and are quoted herewith: (19)

"(a) Except as otherwise provided by this regulation, the quantity of the contents, in all cases of food, if in package form, must be plainly and conspicuously marked, in terms of weight, measure, or numerical count, on the outside of the covering or container usually delivered to customers."

"(b) The quantity of the contents so marked shall be the amount of food in the package."

"(c) Statements of weight shall be in terms of avoirdupois pounds and ounces; * * *"

"(h) The quantity of the contents may be stated in terms of minimum weight, * * * but in such case the statement must approximate the actual quantity and there shall be no tolerance below the stated minimum."

Scales.

The Federal regulations covering the weight of packaged foods, included specifications for the kind of scales to be used in determining weight. Those specifications are in part as follows:

"I. The following specifications have been selected to represent good commercial practice in packaging granular, solid, free-flowing food products:

1. That packages of 5 pounds and below be weighed net; larger packages may be weighed gross and tare.

2. That the following types of scales be used:

For packages of 5 pounds and below: Even arm balance, 1-pound beam, $\frac{1}{4}$ -ounce graduation.

For packages over 5 and under 25 pounds: Even arm balance, 2-pound beam, $\frac{1}{4}$ -ounce graduation.

For packages of 25 pounds and above: Platform scale, 50-pound beam, $\frac{1}{4}$ -ounce graduation.

3. That scales meet the tolerances prescribed in Bureau of Standards Circular 61 and be used at 20 per cent or more of their capacity for packages of less than 25 pounds and at 10 per cent or more of their capacity for larger packages. Tare weights may be made on the same scale as the gross weights.

4. That the exact balance be estimated to within one-half of the sensibility reciprocal of the scale and the poise be set to within one-fourth of the minimum graduation on the beam.

VARIATIONS AND TOLERANCES.

In as much as it is not possible for a man under commercial conditions to accurately weigh a large number of packages (sacks of flour), it become necessary to determine the extent and frequency of the variations, nad to establish tolerances that would be applicable and reasonable under good commercial practice conditions.

Variations have been determined and tolerances established as follows: (19)

"(i) The following tolerances and variations from the quantity of the contents marked on the package shall be allowed:

(1) Discrepancies due exclusively to errors in weighing, measuring, or counting which occur in packing conducted in compliance with good commercial practice."

"(3) Discrepancies in weight or measure, due exclusively to differences in atmospheric conditions in various places, and which unavoidably result from the ordinary and customary exposure of the packages to evaporation or to the absorption of water.

Discrepancies under classes (1) and (2) of this paragraph shall be as often above as below the marked quantity. The reasonableness of discrepancies under class (3) of this paragraph will be determined on the facts in each case."

Runkel (13) has summarized his investigations on variations and tolerances in part as follows:

"(2) The maximum errors of good commercial practice given in Table 15 have been calculated by the use of methods of least squares, the Bureau of Standards tolerances on scales, and the specifications of good commercial practice.

Table 15.—Calculated Maximum Errors of Good Commercial Practice.

SIZE OF PACKAGE	On single packages	On the average of a representative sample	On single packages	On the average of a representative sample
	ounces	ounces	ounces	ounces
2-ounce	0.150	0.097		
3-ounce150	.097		
4-ounce150	.097		
8-ounce150	.097		
1-pound213	.106		
2-pound360	.145		
3-pound363	.148		
4-pound409	.194		
5-pound409	.209		
10-pound544	.373	0.674	0.414
25-pound	5.41	2.68	7.49	3.34
50-pound	5.41	2.68	7.73	3.57
75-pound	5.65	2.92	7.73	3.57
100-pound and above ..	5.65	2.92	8.29	4.13

3. A study of machine weighing has removed practically all doubts of the ability of properly operated machines to meet the calculated maximum errors of hand weighing.

4. Data have been collected which show that the calculated maximum errors represent a close approximation to the maximum variations of good commercial practice. * * *

There are several factors which must be reckoned with when considering the variations and tolerances to be allowed in the weight of packaged food (flour). Runkle (13) has classified errors as *constant* and *variable*. The constant errors are those introduced into every package, and include the errors on ratio, on beam, and on counterpoise weights, on both gross and tare weighing. The variable errors are those due to manipulation of the scale and include the setting of the poise and the balancing of the beam for both gross and tare weighing, (referred to by Runkle (13) as "one-half minimum graduation" and "one-half sensibility reciprocal"). In order to show the influence of each separate factor, and the effect of the several factors in combination, the following tabulation has been made:

Note: The following statements have been compiled from "Table 6.—Calculated maximum errors of good commercial practice (gross and tare weighing)" on page 9, of U. S. Dept. of Agr. Bul. 897 (13).

	Size of Package, pounds.			
	25	50	75	100**
Maximum capacity of scales specified, lbs..	250	500	750	1000
Capacity of beam, lbs.	50	50	50	50
Minimum graduation, ounces	4	4	4	4
No. of packages in representative sample..	10	10	10	10

Maximum constituent errors introduced as read from tolerance tables and specifications.

Constant errors introduced into every package.

	Size of Package, pounds.			
	25	50	75	100**
On tare,				
(a) On ratio, ounces	0.0	0.0	0.0	0.0
(b) On beam, ounces	1.0	1.0	1.0	1.0
(c) Counterpoise weights times ratio of scales, ounces	0.0	0.0	0.0	0.0
On gross,				
(d) On ratio, ounces	0.0	0.5	0.5	1.0
(e) On beam, ounces	1.0	1.0	1.0	1.0
(f) Counterpoise weights times ratio of scales, ounces	0.0	0.69	0.69	1.37
Variable errors due to manipulation of scale.				
On tares,				
(g) $\frac{1}{2}$ minimum graduation, ounces	2.0	2.0	2.0	2.0
(h) $\frac{1}{2}$ sensibility reciprocal, ounces	4.0	4.0	4.0	4.0
On gross,				
(i) $\frac{1}{4}$ minimum graduation, ounces	1.0	1.0	1.0	1.0
(k) $\frac{1}{2}$ sensibility reciprocal, ounces	4.0	4.0	4.0	4.0
Calculated maximum error on single package.				
(a) ² (b) ² (c) ² (d) ² (e) ² (f) ²				
(g) ² (h) ² (i) ² (k) ² (*)	7.49	7.73	7.73	8.29
Calculated maximum error on the average weight of a representative sample.				
(a) ² (b) ² (c) ² (d) ² (e) ² (f) ²				
(g) ² (h) ² (i) ² (k) ² (*)	3.34	3.57	3.57	4.13

** Note: 100 pounds and above.

*Note: Results expressed in ounces.

EXEMPTIONS.

The term exemptions as commonly used, applies only to those packages which are too small to be labeled. In the case of weight the following Federal regulation has been issued: (20)

"(j) A package containing one-half avoirdupois ounce of food or less is 'small' and shall be exempt from marking in terms of weight."

In a broader sense, the term, Exemptions may be construed to include those packages whose weight conforms with the specifications, variations and tolerances heretofore described.

DISCUSSION.

Having shown what constitutes good commercial practice, and the specifications, variations, tolerances and exemptions provided for under the Federal law, it will be of interest to cite a few things in connection with the Texas law and its enforcement.

One mill was indicted on 48 counts, in which it was alleged that there

was an average deficiency in weight of approximately three ounces in each of 48 sacks, whose net weights were stated to be 48 pounds. No consideration or credit by average weights of all sacks in lot, or otherwise, was given to the flour in question.

The Federal regulations provide that the average weight (13) of the sacks or packages in a given lot shall be taken, and specifies that variations "shall be as often above as below the marked quantity." (13)

No consideration was given to moisture content, although practically all investigators recognized moisture as a variable factor likely to affect the net weight of flour, etc., and it is specifically provided for in the Federal regulations, which state:

"(3) Discrepancies in weight or measure, due exclusively to differences in atmospheric conditions in various places, and which unavoidably result from the ordinary and customary exposure of the packages to evaporation or to the absorption of water."

"* * * The reasonableness of discrepancies under class (3) of this paragraph will be determined on the facts in each case."

It has also been developed by investigation into the subject, that the State Weights and Measures officials have recognized the U. S. Bureau of Standards Specifications and Tolerances for Scales—but it does not appear that they have permitted similar variations and tolerances in the weights of the products weighed on those scales, and which, when inspected, were found to conform with the specifications and tolerances established by the afore mentioned Bureau. It rather appears, that the State Inspectors have assumed that the tolerances for a given load on Class A Scales, (22) should apply, rather than the special tolerances provided for the type of scales generally in use in the plants where the flour is packed. Runkel (13) has investigated the types of scales in general use for weighing a certain size package, and has recognized certain capacity scales to be used in weighing certain sized packages as conforming with good commercial practice, namely:

"For packages of 25 pounds and over: Platform scale, 50-pound beam, one-fourth pound graduation.

3. That scales * * * be used at 20 per cent or more of their capacity for packages of less than 25 pounds, and at 10 per cent or more of their capacity for larger packages. Tare weights may be made on the same scales as the gross weights.

4. That the exact balance be estimated to within one-half of the sensibility reciprocal of the scale, and the poise be set to within one-fourth of the minimum graduation on the beam."

The writer has found (4) that the type of scales in common use in several Texas flour mills, conforms to the specifications of good commercial practice as described by Runkel. (13)

The specifications quoted above would easily admit of larger variations in the weight of individual bags of flour—than were complained of in many

of the indictments returned against Texas millers. The tolerances on Class A type of scales recognized by Runkel (13) as conforming with good commercial practice, are covered by the following *proviso* contained in the "Manual for Weights and Measures Officials," (22) published by the U. S. Bureau of Standards, and in general use by weights and measures officials throughout the country. The proviso also appears in Circular 61, (21) referred to elsewhere, and by inference in the Weights and Measures Laws of Texas and of the United States.

"Tolerances. The tolerances to be allowed in excess or deficiency on all platform scales, * * * shall not be greater than the values shown in the following table: * * * And provided further, That these tolerances on all these platform scales **shall in no case be less than the value of one of the minimum graduations on the beam, * * ***"

Accepting Runkel's (13) statement as to the type of scales that conforms with good commercial practice, and applying the tolerances provided for such scales by the U. S. Bureau of Standards, we find that a tolerance of at least 4 ounces (hte minimum graduation on the beam) should be allowed in the net weight of flour, due to htis one source of error in weight alone.

Any material change in the moisture content of flour must of necessity affect the net weight. Modern millers endeavor to maintain a moisture content of 13.50 per cent in their flour—the maximum amount pennitted by pure food laws. The average moisture content of flour after storage is considerably lower than 13.50 per cent. Sanderson (14) has given 11.00 per cent as a fair average moisture content. Henry and Morrison (8) give the average moisture content of wheat flour as 12.30 per cent. We may reasonably expect flour containing 13.50 per cent moisture to lose from one to two and one-half per cent moisture under ordinary conditions of storage. On a 48 pound sack of flour the loss in weight due to moisture would be from one-half to one and one-quarter pounds. Under extreme conditions as much as two to three pounds of weight might be lost by a 48 pound sack of flour in the form of evaporated moisture. The amount of loss in the weight of flour is dependent to a large extent upon the conditions of storage.

When the conditions of storage are favorable, heavy losses in weight may occur. Add to this the variations which may occur in weights and weighing, and variations of one to three pounds per 48 pound sack are easily accounted for.

SUMMARY.

1. Variations in the weight of flour may be caused by (a) changes in moisture content, (b) inaccurate scales and weights, and (c) inaccurate manipulation of scales.

2. The extent of the variations due to changes in moisture content

depend upon (a) the conditions of storage, (b) relative humidity, and (c) original moisture content.

3. The extent of variations likely to result from the errors in the packing and weighing of flour, conducted in accordance with good commercial practice, have been computed and found to agree quite closely with the conditions as found.

4. The advantage of specifying the amount of dry matter content, rather than the weight of a sack of flour, would afford a more reliable basis for enforcement of the Net Weight provisions of Weights and Measures Laws.

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Abstracts

FROM EXPERIMENT STATION RECORD.

"The Kjeldahl Nitrogen Method and its Modifications," A. E. Paul and E. H. Berry, (*Jour. Assoc. Off. Chem.*, 5 (1921), pp. 108-132).—In this report of an extensive investigation of the details of the Kjeldahl nitrogen method, made with a view to perfecting the technique so that discrepancies in results may be avoided, the plan of investigation consisted first in a study of the influence of the various parts of the necessary apparatus, using only pure salts of ammonia for the determinations. This was followed by a study of the details of the digestion, particularly with respect to the influence of the different substances used to hasten oxidation. In this study both ammonium salts and samples of cottonseed meal were used. The method was then studied in detail on the samples of cottonseed meal and on other products, and finally a study was made of the effect on the results of the presence of nitrates in the substances to be digested and of nitrous acid fumes in the atmosphere surrounding the digestion apparatus. Taken in order, the various points brought out in this investigation are as follows:

The Kjeldahl connecting bulb is much more efficient than plain bent tubes with or without bulbs and should be used to obtain the best results. When sufficient acid was used in the receiving flask to neutralize 85.5 per cent of the ammonia distilled over, the amount was 99.87 and 99.68 per cent. This is thought to indicate that, while advisable, it is not absolutely necessary to use sufficient acid to neutralize all the ammonia to be distilled over. Practically all the ammonia passes over with the first 75 cc. of the distillate and all of it with 100 cc.

The volume of the liquid in the distilling flask should be kept well above 10 cc. and the flame never allowed to touch the flask above the liquid. To safeguard against this danger the use is recommended of asbestos guards with holes of such a size that only that part of the flask which actually contains the liquid is exposed to the flame.

If mercury is used during the digestion and sulphid is not added to precipitate the mercury before distillation, a loss of from 12 to 15 per cent of ammonia results. When copper sulphate is used in place of mercury the addition of potassium sulphid is unnecessary. Potassium permanganate is of no value and tends to result in a loss of ammonia. It is impossible to get maximum results with sulphuric acid alone. The reagents in addition to sulphuric acid which yield the maximum results in the minimum of time are combined potassium or sodium sulphate, mercuric oxid, and potassium sulphid. If copper sulphate is used in place of mercuric oxid and potassium sulphid the time of digestion must be lengthened.

Detailed directions embodying these findings are given for the nitrogen determination of cottonseed meal. If mercuric oxid is used, the digestion

should be continued for 3 hours, and if copper sulphate for 6 hours after the solution clears. The time required for the digestion of other materials with mercury and with copper sulphate, respectively, is given as 1 and 2 hours for flour, $\frac{1}{2}$ and 2 hours for powdered milk, 2 and 6 hours for gelatin, 1 and 5 hours for egg albumin, and 4 and 8 hours for tankage.

The presence of nitrates in the original sample was found to yield higher results than would be obtained without the nitrate. The addition of nitrates to the digested solution or the presence of nitrous fumes in the hood in which the digestion was being conducted was found to cause a decided loss of nitrogen. The importance is emphasized of eliminating and carefully guarding against the presence of nitrous fumes in the vicinity of the digestion apparatus.

"The Micro-Nitrogen Determination in Agricultural Materials," W. Geilmann, (*Jour. Landw.*, 68 (1920), No. 4, pp. 235, 254, figs. 4).—This is a brief discussion of the applicability of the micro-Kjeldahl method to the examination of agricultural materials and of the principal sources of error in determination and means of avoiding them.

"Boric Acid For Neutralizing Ammonia in Nitrogen Determinations," H. D. Spears, (*Jour. Assoc. Off. Agr. Chem.*, 5 (1921), No. 1, pp. 105-R).—A comparison has been made on 60 laboratory samples of feeding stuffs of the use of sulphuric acid and of boric acid, as recommended by Scales and Harrison (*E. S. R.*, 42, p. 802), in neutralizing the ammonia in nitrogen determinations. Comparable results were obtained with both methods. Bromocresol purple has been found to give good results as an indicator in the determination with boric acid.

"H-ion Concentration Studies on Distilled Water, Physiologic Sodium Chlorid, Glucose, and Other Solutions Used for Intravenous Medication," J. R. Williams and M. Swett, (*Jour. Amer. Med. Assoc.*, 78 (1922) No. 14, pp. 1024-1026).—The authors advance the hypothesis that when fluids of a much higher or lower H-ion concentration than blood are introduced into the circulation at a rate or in an amount in excess of the capacity of the blood to neutralize them reactions characterized by chills and prostration follow. Similarly it is thought that soreness near the site of subcutaneous injections may be due to localized temporary tissue acidosis or alkalosis. This theory is based on clinical observations and on H-ion concentration determinations of solutions, the use of which has led to such reactions. The substances reported on in this paper are distilled water, glucose, and physiological salt solutions.

Data are presented showing that distilled water if not carefully prepared, or after being stored as a stock solution, becomes sufficiently acid that when used as a solvent it may produce a solution of a much higher H-ion concentration than that of the blood. Glucose solutions become acid when boiled or autoclaved or when allowed to stand for a few hours. Physiological salt solution when prepared with stock distilled water or with

an impure salt may also be acid. It is shown that these solutions may be easily corrected to the proper H-ion concentration by the addition of buffer salts.

"A Study of the Details to the Crude Fiber Method," G. L. Bidwell and L. E. Bopst (*Jour. Assoc. Off. Agr. Chem.*, 5 (1921), No. 1, pp. 58-70).—A critical study of the Official method of determining crude fiber is reported, together with new details for conducting the determination, which, if followed carefully, is said to yield concordant results. The more important points brought out in the study are summarized as follows:

"Liebig condensers are the most efficient that may be used. Samples taking more than 5 minutes to filter after digestion should not be reported. Samples of high protein content, under delayed filtration, act just opposite to those of low protein content. Any container having 1.5 in. of boiling depth may be used. The presence of fat in charge during fiber determination noticeably raises the results. There is a gain in ether extract when the charge has been previously digested with 1.25 per cent sulphuric acid. The finer the material is ground the lower the fiber content. More uniform results are obtained using a 40-mesh sieve."

"The Determination Of Carbon Dioxid In Baking Powder," C. S. Robinson and S. L. Bandemer (*Jour. Indus. and Engin. Chem.*, (1922), No. 2, pp. 119).—The method of determining carbon dioxid previously noted (*E. S. R.*, 44, p. 203), which has been found practical for determining total CO₂ in baking powder, has been extended to the estimation of residual CO₂ in baking powder. The technique for total CO₂ is the same as that originally described for its determination in limestone except that 100 mg. samples are used. For residual CO₂ a 2-gm. sample of the well-mixed material is weighed into a small beaker, 22 cc. of distilled water is added, and the mixture is thoroughly stirred and allowed to stand for 20 minutes at room temperature and for the same length of time in a bath of boiling water. It is then boiled for 1 minute and diluted to 25 cc. in a volumetric flask, 1-cc. samples being used for the determination.

A Quick Method of Determining Protien in Wheat

(Reprinted from National Miller, December, 1922)

By P. H. BIMMERMAN and W. L. FRANK.

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One of the principal objections to the use of the Official Method of Kjeldahl Nitrogen Determination in wheat classification by flour mills, is the length of time required to make a test. Under ordinary working conditions 2½ to 3 hours are required with the official method, and sometimes longer. The authors have made comparative tests between the Official

Gunning Method and a modification of same using varying amounts of 60% perchloric acid (HClO_3).

The Role of Perchloric Acid.

Perchloric acid is a powerful oxidizing agent and serves to reduce the time required for digestion. (Oxidation of the organic matter present in the sample.) There are certain other advantages of the modified method, which will be discussed.

Several Precautions Must Be Taken.

Our preliminary work indicated that the amount of the several chemicals used in each test affected the results. Definite amounts of sodium sulphate, sulphuric acid, copper sulphate, perchloric acid, and of sample were found to be important considerations. Temperature (size of flame) and length of heating period before and after clearing, also affected the results. Our observations lead us to conclude that,

1st. The following amounts of chemicals should be used for a 2 gram sample

- | | |
|---------------------------------|------------------|
| (A) Sulphuric acid sp. gr. 1.84 | 20cc. |
| (B) Sodium sulphate-anhydrous | 3 to 4 grams. |
| (C) Copper sulphate-crystals | 0.1 to 0.2 grams |
| (D) Perchloric acid—60%, 4cc. | 0.2 grams |

2nd. The flame should be turned very low during first minute—so that it covers an area on flask not larger than a quarter. It may then be turned up until it covers an area as large as a silver dollar.

3rd. That temperature (flame) should be so regulated that the contents of flask will clear in approximately 8 to 10 minutes. Heating should be continued for 10 minutes after contents of flask clear. Care should be taken not to turn flame so high that it will strike the side of the flask above the liquid.

Amount of Perchloric Acid vs. Time vs. Protein.

Table No. 1, presents the results of some of our preliminary tests which show the effect of varying amounts of perchloric acid upon both the time required for digestion and the protein result.

Preliminary tests—part of which are reported in Table No. 1—indicated that the most consistent checks were obtained when 0.20 grams CuSO_4 , 20cc H_2SO_4 , 3-4 g. Na_2SO_4 , and 4 cc. HClO_3 were used and the heating so regulated as to require from 8 to 10 minutes to clear, and 18 to 30 minutes to complete oxidation of the organic matter present in a 2 gram sample.

Market run samples were used in this comparison without any attempt at selection. The first official determinations were all made by one of the authors and the official checks were all made by the other author. The perchloric tests were made jointly by both authors. The same solutions and reagents were used throughout. Averages of results may well be re-

garded as a fair basis of comparison of methods on samples of varying protein content. Results are summarized in Table No. 2.

From Table No. 3 it is apparent that correct results (within 0.25 percent of accepted correct results by the official Gunning Method) can be obtained as frequently by the perchloric acid method as by the official Gunning method. (Note: In all cases where the difference between results obtained by these methods exceeded 0.25%, the tests were repeated in duplicate by both methods. The results so obtained were accepted as correct. An error was charged to the method which varied most from the correct results on the first test, and to both methods if neither was within 0.25 per cent of the correct results.)

TABLE No. I.

The effect of varying amounts of chemicals used, upon time required to complete a test and upon the protein result.

Size of sample	Amounts of Chemicals used				Time required to		Protein NX5.7
	copper	H ₂ SO ₄	Na ₂ SO ₄	HClO ₃	Clear	complete	
2 grams	0.05 (*)	25cc.	3-4 g.	3	28.0 min.	43.0 min.	13.02
3 grams	0.05 (*)	25cc.	3-4 g.	4	18.0 min.	33.0 min.	13.44
2 grams	0.05 (*)	25cc.	3-4 g.	5	6.5 min.	21.5 min.	11.31
2 grams	0.2 (**)	25cc.	3-4 g.	3	43.0 min.	60.0 min.	13.40
2 grams	0.2 (**)	25cc.	3-4 g.	4	18.5 min.	34.0 min.	13.40
2 grams	0.2 (**)	25cc.	3-4 g.	5	6.0 min.	21.0 min.	12.72
2 grams	0.2 (**)	15cc.	3-4 g.	2	45.0 min.	55.0 min.	13.48
2 grams	0.2 (**)	20cc.	3-4 g.	5	6.5 min.	16.5 min.	8.10
2 grams	0.2 (**)	20cc.	3-4 g.	4	10.5 min.	20.5 min.	13.44
2 grams	0.2 (**)	25cc.	3-4 g.	4	13.0 min.	23.0 min.	13.40
2 grams	0.2 (**)	20cc.	3-4 g.	4	8.0 min.	18.0 min.	13.45
2 grams	0.2 (**)	20cc.	3-4 g.	4	8.0 min.	23.0 min.	13.52
2 grams	0.2 (**)	20cc.	3-4 g.	4	8.0 min.	28.0 min.	13.50
2 grams	0.2 (**)	20cc. (Official Gunning Method)					13.44

Notes: 1. (*) Copper wire used in these tests.

2. (**) Copper sulphate crystals used in these tests.

From the data given in Table No. 4 it appears that the results on two portions of the same sample of wheat when analyzed by the official Gunning method, may be expected to vary more than when the same portion is analyzed by the official Gunning and perchloric acid methods.

Further Observations.

There are other advantages in using perchloric acid in the determination of protein. The amount of (Na₂SO₄) Sodium sulphate may be reduced to 3 to 4 grams per test, likewise the length of time required for distillation and the amount of gas consumed per test are relatively less. When the

proper amount of chemicals is used, there is less danger of foaming during distillation than is true of the official method.

TABLE No. 2.

A comparison of methods of analysis vs. protein results.				
No. of samples	Range in protein (off check)	Average protein contents		
		First official	Perchloric	Official check
4	13.00 and over	13.43	13.59	13.61
20	12.00 to 12.99	12.33	12.21	12.23
15	11.00 to 11.99	11.65	11.69	11.72
3	10.00 to 10.99	11.07	11.03	10.93
42	10.00 to 14.10	12.10	12.07	12.08

This permits very rapid distillation of the liquid. It is advisable to use at least 300 cc distilled water in diluting liquid contents of flask before distillation.

Special precautions that should be taken:

1st. Do not use over 4 nor less than 3 grams Na_2SO_4 .

2nd. Shake contents of flask until thoroughly mixed after adding approximately 250 to 300 cc distilled water and before adding concentrated caustic NaOH to prevent violent chemical action of the heavy liquids that would be in bottom of flask.

TABLE No. 3.

Comparison of accuracy of results, official check method vs. perchloric acid method.

	Perchloric Method	Official check Method
Number of samples analyzed.....	42	42
Number samples on which analyses varied over 0.25%	7	7
Numbers of samples on which a second test on sample showed first results were incorrect....	4	6
Average percent protein of 42 samples.....	12.07	12.08

3rd. Do not add zinc until after dilution of liquid contents of flask.

4th. Distill for 20 to 25 minutes, having contents of flask boiling as rapidly as conditions will permit.

Conclusions.

1st. A substantial saving in time can be effected by using perchloric acid in protein determinations on wheat.

2nd. Slight variations occur in results on individual tests as compared with official tests.

3rd. The difference in results on the basis of average results of 40 or more cars is negligible.

4th. For the purpose of grain classification and routine mill control

TABLE No. 4.

Comparison of accuracy of perchloric acid method vs. official check method.		
Number of samples on which results were:	First official method	Perchloric acid method
(a) Below official check method.....	17	23
(b) Above official check method.....	19	12
(c) Identical with official check method.....	6	7
Average variation of results below the official check method in per cent.....	0.290	0.125
Average variation of results above the official check method in per cent.....	0.262	0.136
Average variation of results from correct official check method results in per cent.....	0.236	0.107

(Note: The perchloric acid results were obtained on a portion of the sample after grinding, on which the official check method results were obtained. The results for the first official method were obtained on a portion of the same 1000 grams of wheat as for the official check method, but each portion was ground separately.)

work, the perchloric acid method offers several advantages of considerable practical value.

5th. The above statements are based upon actual data on market run wheat, and while rather limited in scope, they at least suggest the need of exhaustive investigation of the method. Our results have been very encouraging thus far.

The authors will be glad to hear from other chemists who have used perchloric acid in protein determination.

Survey of Colorado Wheat Crop for 1922-1923

By H. D. LIGGETT, Jr.
Colorado Milling and Elevator Company

For many years the reputation of Colorado Wheat has not been very high; that is, in certain sections of the country. Many millers, grain dealers, bakers, etc., think that Colorado Wheat is either soft and white or of the yellow-berry variety. We admit that some of our wheat is soft and white, and some is yellow-berry. But our soft white wheat is ranking very high in the Southeast as a pastry and biscuit flour; and our farmers, by scientific farming and crop rotation, have overcome, to a great extent, the yellow-berry

in Colorado Wheat. We will have considerable yellow-berry—but so have Kansas, Nebraska, Minnesota, and other states.

A great deal of work has been done, and is being done, at the Government Experimental Station at Fort Collins, under the direction of Mr. Headdon. We owe a great deal to Mr. Headdon for his work and research done on Colorado Wheat. (He has studied the climate, soil, plant food, and minerals which affect our wheat, and has started one of the best white wheats that Colorado has grown.)

In years past the wheat in Colorado was mostly of a white variety, but now the farmers of the state have begun to grow the Turkey Red variety as a Winter Wheat—and the Marquis, as a Spring. The farmers of Colorado thought that the climate was such that a Turkey Red could not yield enough to make it profitable. But they began to experiment with different wheats and finally came to the conclusion that a Winter Wheat would yield more and really pay more than any other variety. So now in Colorado the wheats that predominate are of the Hard Wheat variety; and the White Wheat is gradually being relegated to the background.

Colorado is divided into several sections where wheat has to be grown under different climatic and soil conditions. We have the irrigated sections, and we have the dry land sections. In some sections the winters are more severe than in others; and, above all, the soil and its mineral constituents play the big part. Of course, the important factor is the ratio of the Nitric Nitrogen to the Potassium which can be utilized. In certain of our localities the predominance of the Potassium causes that locality to produce a mealy wheat, which could really be called yellowberry; and any wheat, no matter how good when introduced as seed, will degenerate gradually to the character of the wheat in that locality.

Irrigation plays a big part in the farming of our Colorado land. We find that a land which, under dry cultivation, will only produce a yield of from 10 to 20 bushels per acre, will, under irrigation, produce from 20 to 54 bushels per acre. A great many authorities claim that wheat of the same grade and variety will, when grown under irrigation, give a flour identical in every way with that of the same wheat grown on dry land. Our tests here show that a dry land wheat generally has a higher protein content and makes a stronger flour.

This last year in Colorado, our irrigated farms were short of water; the farmers were not able to put on as much water as they ordinarily would. Consequently, we find that the yield per acre went down, but a much harder and darker variety of wheat was grown. This wheat shows a high protein content—some of it going as high as 16.35%; and other wheat from practically the same locality, but which received more water, falling as low as 11.5%. We find that this high-protein wheat can stand a longer tempering period and a little more water than in previous years. We find that, except in one or two sections, our crop this year in Colorado is quite free from

smut, rust, and other diseases; but owing to carelessness or misfortune, there is considerable stack-burned wheat in some localities.

We find that our eastern and southeastern Colorado dry land wheat is practically the same as the western Kansas Turkey. It, being grown on fallow soil, gives a high protein test and makes a very good flour. In many instances the wheat kernels are small and slightly shrunken—probably due to the excess Nitrogen and lack of water.

In past years, one of the principal reasons for the yellow-berry in Colorado has been the planting of wheat year after year on the same land, especially in the dry land sections; but the crop rotation has overcome that difficulty, so that now our dry land wheat is a very good grade of wheat.

Our Marquis Wheat crop this year has been a very good one. The berries have, as a rule, been very plump and vitreous; many sections produced around 62 pounds. This wheat makes a very good flour, which has a ducing wheat that ran as high as 15.5% in protein, and having a test weight strong and fairly elastic gluten. This wheat does not seem to give the volume in bread that the Turkey does, but it gives a beautiful, close-grained texture and very white loaf of bread. A small percentage of Marquis blended with the dry land Turkey gives a whiter flour than the straight dry land; and gives a whiter loaf of bread. The Marquis this year seems to be better, because of the lack of water in the irrigated sections.

Several new varieties of wheat have been introduced into Colorado recently. The Early Baart, from the Northwest, is having only mediocre success when grown in Colorado. The Kitchener Marquis seems to be having fairly good success, and it is now being grown in the San Luis Valley. Some of our wheat grown on the Western Slope of Colorado, namely, the Black Hulled Turkey, took second prize at the Grain Exposition at Chicago. It is a large berry and makes a very good flour. More experimenting is now being done with the Winter Wheats than there is with the White Wheats.

Kansas Chemists Round Table

FOURTH MEETING, DECEMBER 2, 1922,

The fourth meeting of the Millers' Chemists' Round Table Club held in the Goerz Club rooms at Newton, Kansas, on December 2, 1922, surpassed any meeting yet held. More actual good was accomplished and more helpful suggestions given. The Chemists went home feeling that some real results had been obtained.

The attendance was about the same but the area represented was larger. Twenty chemists and three mill superintendents attended. Topeka,

Winfield, Arkansas City, and Alva, Oklahoma, were represented as well as the cities more centrally located. The chemists are glad to have the mill superintendents present and urge them to attend and give their suggestions.

Quite a number of chemists brought their loaves of bread, baked from 95% flour and a discussion of these loaves was the start of the program. The object was not to discuss the quality of the flour but the type of loaf and treatment of the dough and bread. Each chemist in turn was asked to tell why he baked the type of loaf represented and what treatment he gave the dough. Opinion was unanimous that the loaf brought by Ross of Lindsborg was the best and he gave his method in detail, as follows:

Formula:—340 gms. flour, $1\frac{1}{2}$ percent salt, 3 percent sugar, $1\frac{1}{4}$ percent yeast, 8 gms. lard, mixed in Hobart cake mixer for 2 min. 30 sec. Fermented at 80 degrees F. 2 1-3 hours first punch, 50-55 min. second punch, 20-25 min. third punch. Bench 10 min. and pan. Proofed 50 min. at 90-95 degrees F. Baked 28 min. at 480 degrees —420 degrees F.

Wet rags were kept over crocks during fermentation and steam was used in cabinet during proof and in oven during bake.

The type of loaf to be desired was one having a fine, close, soft, silky texture, even white color, thin soft golden-brown crust, and regular shape not broken only on one side. Steam used during proof and bake is one of the main factors in obtaining such a loaf and very few chemists are equipped for such procedure. Method of molding is another important factor.

After the bread discussion the meeting was again called to order by Mr. Schultz and the minutes of the last meeting were read.

J. R. Hess reported on the protein results obtained on sample of flour sent out by him. The flour methods of determining protein were as follows:

Method 1. Kjeldahl-Gunning Method using copper as catalyst and zinc as ebullition agent.

Method 2. Same as Method 1 except using pumice or other inert agent as ebullition agent.

Method 3. Kjeldahl Method using a mercury catalyst and any ebullition agent.

Method 4. Using sodium or potassium sulphate alone without any catalyst and using zinc as ebullition agent.

There was very little variation in results obtained by each individual chemist between the different methods. The largest variation being obtained using the method most unfamiliar to the chemist. So the variation between chemists did not appear to be in the methods used so much as in the standardization of solutions. However, two of the above methods are undesirable. The largest variation in results was obtained in Method 1, the undesirable feature being the probability that copper and zinc occurring together may act as a catalyst to break down nitrates into ammonia. Method 4 is undesirable because of the length of time required for digestion.

Hess suggested that the methods of standardization of solutions should be made more uniform.

L. C. Mann suggested a Barium chloride method for standardizing sulphuric acid solution and J. R. Hess suggested the use of benzoic acid to standardize the caustic soda solution. R. J. Clark mentioned that succinic acid had been used very successfully in standardization but that the water of crystallization of succinic acid had been objected to by the Bureau of Standards.

Motions were made and passed that Hess draw up the Benzoic Acid method and Mann the Barium Chloride method and that the secretary incorporate the two methods in the minutes of the meeting so that they may be tried out by the members.

Barium Chloride Method for Sulphuric Acid.

Draw off 25 cc. H_2SO_4 solution, add 5 cc. of a solution made by putting $2\frac{1}{2}$ c.c. conc. HCl in 57 c.c. H_2O , and then add 50 c.c. H_2O . Heat to boiling and add 20 c.c. boiling Ba Cl_2 solution (1.62 gms. BaCl_2 in 90 c.c. H_2O) all at once with constant stirring for two or three minutes, then let stand. At end of a half hour (after testing for complete precipitation) decant liquid through filter. Wash precipitate by decantation with hot water and subsequently upon the filter with hot water until filtrate is free of chlorine. Dry, ignite, and weigh (Method is taken from George McPhail Smith's Quantitative Analysis. Author claims there are unavoidable errors with BaSO_4 method but that by using concentrations specified, the errors are kept to a minimum and compensate each other).

Benzoic Acid Method for Caustic Soda.

Observe the following precautions:

1. The acid should be carefully fused before use in a covered glass or platinum vessel placed in an air-bath. The temperature during fusion must not rise above 140 degrees C.; it is best to keep it below 130 C. and to cease heating as soon as fusion is complete. (Fusion is unnecessary unless a high degree of accuracy is desired. Titrations carefully made check to 1-2 parts in 10,000 and consistent differences as great as 6-7 parts in 10,000 have been observed between fused and unfused materials which have stood in the laboratory for 1-2 years).
2. The acid should be dissolved in alcohol (about 20 c.c. for a 1-gram sample) and a blank test made upon the same volume of alcohol so that the necessary correction may be applied in the subsequent titration.
3. The titration must be made in the cold with carbonate-free alkali and the necessary precautions taken to prevent the access of carbon dioxide from the air during titration. Phenolphthalein is

the most satisfactory indicator, and 75 c.c. is a convenient volume in which to make the titration.

R. J. Clark reported that the method for determining protein used at the Manhattan Agricultural Station was the Kjeldahl-Gunning Method using sodium sulphate with copper as catalyst and zinc as ebullition agent. Additional features were the use of 25 c.c. conc. H_2SO_4 instead of 15 or 20 c.c., the digestion for 45 min. after becoming clear, and the use of N-5 H_2SO_4 and N-14.01 NaOH with methyl red indicator. The fact that proteins should be digested longer than just to a clear green deep color was emphasized.

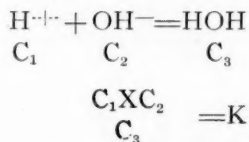
Mr. Mann discussed the effect of the type of flame and the shape of rack upon the quantity of protein obtained and considered the loss using gas turned up too high.

The main address of the afternoon was given by R. J. Clark on Hydrogen ion. A short historical sketch of the dissociation theory was first given carrying it down through Arrhenius, Uernst, Pasteur and Oswald. There are two kinds of methods for determining the H-ion content—the colormetric and electrometric. The advantages of the electrometric over the colormetric are:

1. Turbidity has no effect on the determination.
2. One apparatus covers the complete range from normal acid to normal alkali.
3. Greater precision is obtained.
4. No titrations are necessary.
5. It is possible to titrate to any given pH.
6. No other method shows up changing acidity as well.
7. Colormetric methods are based on electrometric methods, so the basic method might as well be used.

The one advantage of the colormetric methods over the electrometric is that they are more rapid.

Mr. Clark then gave a more detailed account and blackboard demonstration of the dissociation theory and law of mass action with the following formulae:



$$\text{C}[\text{H}^{+}] + \text{C}[\text{OH}^{-}] = K(\text{C}[\text{H}_2\text{O}]) = 10^{-14} \text{ at } 22 \text{ degrees C}$$

He discussed electrochemical formulae dealing with electrode potentials

$$E = E_0 + \frac{RT}{nF} \log e C$$

E =electrode potential at ionic concentration.

E_0 =electrode potential at molar concentration.

R =gas constant=(8.32 joules per degree).

T =absolute temperature. (273 degrees + Temp. C).

n =valency of ion.

F =faraday=(96500 coulombs).

C =ionic concentration of solution.

E_0 =.277 volt in normal solution.

$$\frac{RT}{nF} \times 2.303 (\text{change log from base } e) = \frac{8.32 \times 298 \times 2.303}{1 \times 96500} = .0591$$

.0591 for H-ion and temperature at 25 degrees C.

Then $E = +.277 + .0591 \log C[H^{+}]$.

Change in electrode potential with concentration is given by the expression:

$$E_0 - E = \frac{RT}{nF} \log \frac{C_1}{C_2}$$

The symbol pH is the log. of hydrogen-ion concentration represented in the above formulae. It is sometimes called the acidity. Roughly it is the exponent of the H-ion concentration.

With $C[H] \times [H]10^{-1}$ and $C[OH] \times [OH]10^{-13}$, $pH=1$.

Clark recommended two books on the subject:

1. "The Determination of Hydrogen-Ion," by Mansfield Clark.
2. "Theory of Indicators," by Prideaux.

The University of California has completed a table giving the pH value for each voltage through a wide range of concentrations.

General discussion of H-ion concentration of flour and its value as a determination factor in the fermentation period and baking qualities of a flour then took place. It was stated that the time is coming when every mill laboratory will have a H-ion apparatus and viscosimeter and the baking qualities of flour and also of the wheat to be used will be determined. Data so far is not very generally known but individual laboratories are installing the apparatus and gaining their own knowledge.

Hess' method for determination of ash was taken up but owing to the few in number who had tried the method it was decided to lay the subject aside till the next meeting and in the meantime more might try it out. Very little objection however could be found to the method. Some agricultural stations have used it for some time in perhaps a modified form.

It was decided to hold the next meeting in Arkansas City some Saturday during the first part of February and to start at 10:00 a. m. and continue

through the day. Mr. McVey was appointed to arrange the details of the meeting in this city. Mr. Leatherock was appointed to check up on moisture determination with vacuum ovens to determine what time, temperature, etc., is to be recommended. He will report at Arkansas City.

It was also decided that each chemist should again bring a loaf of bread baked from his 95 % flour to see if there had been any improvement in baking methods as a result of the comparison at Newton. Other subjects to be discussed will be standard methods of treating doughs, methods of standardization of solutions, and methods of judging quality of gluten.

Adjournment.

H. L. Lentz, Secretary.

Kansas Flour Mill Chemists Meeting

FEBRUARY 10, 1923.

The fifth meeting of the Chemists Round Table will occur on February 10, Saturday, at Arkansas City, Kansas.

The morning session will commence at 10:00 a. m. and the afternoon session at 1:00 p. m. Sessions will be held in the Commissioners' room in the City Building. Luncheon will be served at noon at the Osage Hotel, just a short distance from the City Building.

Those who expect to attend will please notify R. V. McVey, Arkansas City Milling Company.

Remember, Chemists, this is to be an all-day meeting. Come loaded to make this as "peppy" and helpful a meeting as we had at Newton.

Bring your loaf of bread again. We want to discuss the molding of dough and its effect on the loaf, the judging of the quality of gluten from the baked loaf, Hess' method for ash, moisture determination with the vacuum oven, and standardization of solutions.

Chemists Round Table, H. L. Lentz, Secretary.

NOTICE .

An apology is due to Mr. Chauncey Foster, chief chemist of the Hecker-Jones Jewell Company and the Company for omitting the name of the Company from the list of Sustaining Members in the last two issues.

Yours sincerely,

J. R. HESS, Editor.